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SAMPLE SWATCH QUARTERLY

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NEW YORK, OCTOBER 14, 1929

Dyeing Rayon Materials in Black Shades

Property of Cellulose Acetate Rayon—Production of Black Shade—Qualities of Aniline Black on Cotton—Dyeing Black Shades

By A. J. HALL, B.Sc., F.I.C., F.T.I.

IN spite of the present-day demand for dress and other materials in bright, attractive colors, there remains a persistent call for materials dyed black. It is true that this demand has fallen off very considerably since the war, but nevertheless it exists; and because it frequently happens that the dyeing of black is concentrated in the hands of a limited number of dyers, it can form for such dyers an important part of their trade.

The introduction of rayon has made a great deal of difference in the technique of black dyeing; for although cellulose rayons have much the same dyeing properties as cotton, yet their greater affinity for dyestuffs in general necessitates special attention to the details of dyeing processes; and, of course, as is well known, cellulose acetate rayon demands dyeing processes quite different from those employed for cotton or cellulose rayon. These differences demand much consideration in dyeing materials containing mixtures of rayon and cotton or mixtures of different rayons.

Turning to cellulose acetate rayon, it may at once be noted that this gives the greatest trouble in dyeing black. The one direct black dye available for this type of rayon—Celatene Black—has but moderate fastness to light. It is believed that this dye is used to but a limited extent for black dyeing. No doubt much research is being carried on with the object of discovering a perfectly satisfactory direct black dye, but there are no indications of immediate success in this direction. Much more probable is the discovery within the near future of entirely satisfactory methods for producing oxidation blacks on cellulose acetate silk; and as is well known in the case of Aniline Black on cotton, such a type of black can be extremely fast to all agencies.

The following are the chief methods for producing a black on cellulose acetate rayon:

1. Dyeing with an amine, diazotizing, and coupling with a suitable naphthol.

2. Dyeing with a dyestuff capable of producing directly a black shade.

3. Dyeing with a substance capable of being oxidized to black on the fiber.

4. Dyeing with a mixture of yellow, red and blue dyes which can be directly applied.

5. First hydrolyzing the cellulose acetate rayon and then dyeing it by methods suitable for viscose or other cellulose rayon.

Of these various methods there can be no doubt that the first is most largely used, and nearly all dye makers market suitable amines capable of being diazotized and coupled to form a black. Generally these amines are colored amino-azo compounds. Thus the amine which is sold under the name of SRA Black IV gives a brownish orange color on the rayon before diazotization, and Dispersol Diazo Black A Paste first gives a somewhat yellower shade. Although these amines give colors on the rayon, they should not be used for this purpose alone, since these shades usually have but little fastness. After diazotization these amines can be coupled with either *amines* or *naphthols* for the production of the black shade. If reference is made to any book on dyestuffs, say the Colour Index, it will be noticed that black dyes are most frequently obtained by using an amine, such as phenylene diamines, for coupling; naphthols are used to a less extent. It would therefore be imagined that amines would be used in the dyeing black of cellulose acetate rayon. This is not the case, however, for beta-oxynaphthoic

acid is almost universally used. Obviously there must be a definite reason for this.

Property of Cellulose Acetate Rayon

In discovering this reason attention must be drawn to the fundamental property of cellulose acetate rayon by which it is able to absorb (dissolve) amines and naphthols from their aqueous solution or suspension. It is thus obvious that during the coupling the silk may absorb a considerable proportion of the coupling component apart from that which definitely combines (couples) with the diazotized amine. These absorbed coupling components may be persistently retained by the rayon throughout subsequent washing and soaping, so that the fastness of the black developed shade may be affected by gradual decomposition of the absorbed coupling component during storage of the dyed material. Now naphthols are much more permanent and stable than amines, such as meta-phenylene diamine. For instance, if cellulose acetate rayon be dyed with SRA Black IV and coupled with meta-phenylene diamine instead of beta-oxynaphthoic acid, it will be found that the black shade tends to become brownish on storage, due to aerial oxidation of uncoupled amine present in the rayon; if the dyeing be stored between white paper it will be found that the paper may become stained brown for this same reason. Hence, on the whole it is much safer to couple with naphthols, and this is one of the chief reasons why amines are not used in developing black shades on cellulose acetate rayon.

Although the production of a black shade on cellulose acetate rayon by means of a developing dye appears simple, it is by no means difficult to obtain inferior dyeings due to lack of attention to the details of the dyeing process. Some time ago the writer was given a handsome black shade on cellulose acetate rayon fabric as a pattern for matching purposes; the black was obtained by means of a developed black. The pattern was put aside within an envelope for a few weeks, and it was then a matter of great surprise to find that the warp threads had assumed a reddish navy-blue color. On further investigation this change was traced to the influence of ammonia fumes; on exposing the fabric to ammonia it immediately became an unpleasing reddish-black shade. Since that time numerous tests of various cellulose acetate rayon fabrics dyed with developed blacks have shown that quite a considerable proportion are liable to redden on treatment with ammonia. On the other hand, cellulose acetate rayon carefully dyed with, say, SRA Black IV is quite fast to such treatment. It is believed that in the case of blacks which redden the fault is entirely with the dyer rather than the dyestuffs.

One of the main points in the successful dyeing of black developed shades is the allowance of sufficient time for both processes of diazotization and coupling. In no case does either of these processes proceed so rapidly as in the case of developing on cotton materials. If diazotization is incomplete, then during the subsequent process of coupling part of the diazotized amine will couple with the undiazotized portion; and if coupling is incomplete, then the residual diazotized amine will gradually decompose. In either case unstable colored compounds are formed which, being reclaimed by the rayon, reduce the fastness of the resulting shade in addition to impairing its pleasing tone. For these reasons it is preferable to carry out the diazotizing and coupling processes at a temperature slightly higher than is usual, in order that they may be fully completed. Thus diazotization should be effected at 15° to 20° C. and coupling at 50° C. Not less than thirty minutes should be allowed for each process, and the rayon should finally be thoroughly soaped to remove all loosely adhering color and so reduce any possible defect of rubbing.

Production of Black Shade

Undoubtedly the most desirable method of producing a black shade is by means of a direct black dye; but it is found that the one or two dyes of this class available are not completely satisfactory as regards their fastness. More important still is the fact that for the production of a full deep black shade it is necessary to use a very large proportion of dye, and this renders the dyeing process costly. Why is the discovery of a satisfactory direct black dye so long delayed? The answer to this question is difficult to state with certainty, but there is reason to believe that the most important factor is that black dyes are usually of very complex composition and cellulose acetate appears unable to dissolve such complex molecules. For example, the black pigment produced by coupling diazotized Black IV with beta-oxynaphthoic acid is strongly retained by cellulose acetate rayon if produced within the rayon itself in the usual manner, but the rayon will not absorb a colloidal dispersion of this pigment if prepared apart from the rayon. Similarly a black shade can be produced by oxidizing aniline within cellulose acetate rayon, but the rayon will not absorb the black oxidation product prepared outside the rayon —it will not even absorb the intermediate compound emeraldine. Numerous other similar instances could be given and they all appear to support the statement made above that cellulose acetate rayon cannot apparently absorb highly complex molecules such as characterize black dyestuffs. Celatene Black is an anthraquinone compound of but moderate complexity, and this accounts for its absorption.

The third method of dyeing is concerned with oxidation dyes. For various reasons the ordinary copper and prussiate Aniline Black dyeing processes fail to produce Aniline Black on cellulose acetate rayon, and it is doubtful if black shades are being produced

by these methods anywhere. But, on the other hand, it ha been found possible to produce a good black by oxidizing logwood on this type of rayon. This method using logwood is protected by E. P. 263222 and U. S. P. 1657656, and experience shows it to be convenient and satisfactory. The rayon is first dyed at about 75° C. in an aqueous solution of logwood until it acquires a deep brown color; it is then immersed for about one-half hour in a dilute 1% solution of sodium bichromate maintained at 60° C. whereby the brown gradually changes to a deep black. Fortunately, the black shade is somewhat bluish in tone and therefore pleasing. Comparison of the fastness to light of cellulose acetate rayon dyed with SRA Black IV and logwood has shown the former to be considerably faster; but, of course, the logwood method has the great advantage of being more easily and rapidly carried out. It is interesting to note that if cellulose acetate rayon is first dyed brown with logwood and then passed through the usual prussiate Aniline Black dyeing process, the brown shade is not converted to black, as perhaps might be anticipated; it remains unaffected.

Qualities of Aniline Black on Cotton

Because of the excellent fastness qualities of Aniline Black on cotton materials it is but natural to expect that a considerable amount of effort has been devoted to modifying the usual processes so that they dye cellulose acetate rayon equally satisfactorily. So far these efforts have mainly resulted in the adoption of amines other than aniline which lend themselves more readily to oxidation. Thus three years ago the writer (E. P. 258699 and U. S. P. 1657255) found that if 2:4-diaminodiphenylamine or a mixture of aniline and this substance were used instead of aniline alone in the copper Aniline Black process, then fast black shades were obtained. More recently G. H. Ellis, of British Celanese, Ltd. (E. P. 311435), has protected the similar use of para-aminodiphenylamine or mixtures of aniline and this substance. If desirable, these amines can be applied to the cellulose acetate rayon by the methods such as are used for dyeing the rayon by SRA dyes and oxidized subsequently. Of course, a larger production is obtainable if the usual Aniline Black padding and aging method is employed. The following examples show how these processes may be applied:

Cellulose acetate rayon fabric is impregnated with a solution consisting of:

10 parts 2:4-diaminodiphenylamine
3 parts glacial acetic acid
6 parts hydrochloric acid of 28° Tw.
4 parts sodium chlorate
0.5 part copper sulphate

then exposed for five minutes in a warm, moist atmosphere at 70° C., rinsed in warm water, soaped at 60° C., and then rinsed in warm water and dried.

It will be noted that no afterchroming is necessary when the 2:4-diaminodiphenylamine is employed. If an alternative dyeing process is desired, then the following will be found satisfactory:

Ten parts of cellulose acetate rayon are immersed for one hour at 75° C. in a liquor containing:

0.75 part 2:4-diaminodiphenylamine
0.60 part soap
0.60 part ammonia of sp. gr. 0.920
300 parts water

then rinsed in warm water and oxidized by immersion in a liquor for one-half hour at 65° to 70° C. in a liquor containing:

4 parts sodium chlorate
25 parts hydrochloric acid of 28° Tw.
1 part copper sulphate crystals
300 parts water

again rinsed in warm water, soaped, again rinsed, and dried.

By the method of Ellis, cellulose acetate rayon fabric is impregnated with a liquor prepared by mixing together equal volumes of the following solutions A and B:

Solution A—
5 parts para-aminodiphenylamine
5 parts aniline
10 parts 10% lactic acid
30 parts 50% acetic acid
20 parts methylated spirit
30 parts water

Solution B—
5 parts aluminium chloride of 52° Tw.
5 parts chromium chloride of 52° Tw.
0.4 part cupric chloride crystals
6 parts sodium chlorate
54 parts water
30 parts 50% aqueous solution of gum arabic

then dried, aged for about five minutes with moist steam at 100° C., and soaped for fifteen minutes at 80° C. with a ¼% soap solution to remove loose color. Afterchroming is optional.

In considering these special Aniline Black methods it must be noticed that both para-aminodiphenylamine and 2:4-diaminodiphenylamine are more expensive than aniline, and that dyeing costs are further raised particularly in the process of Ellis because of the necessity to use the various organic chemicals specified. There is thus still a demand for a *simple* process, also cheap, for dyeing Aniline Black on cellulose acetate rayon. The writer has recently discovered such a method using aniline alone, but a description of this

process must be reserved for a later article, since it forms the subject of patent applications.

Before leaving the dyeing of oxidation Aniline Black on cellulose acetate rayon, reference should be made to an interesting process by G. H. Ellis (E. P. 298699) in which the rayon is first impregnated or treated so as to absorb suitable amines, and then oxidized for the formation of a black shade by immersion in an oxidizing liquor in which the oxidant is para-toluenesulphonchloramide. In this process it again appears that the amines found most suitable do not include aniline but rather para-aminodiphenylamine and even alpha-naphthylamine (this latter substance gives a brown shade instead of a black).

Dyeing Black Shades

Dyeing black shades by means of mixtures of red, yellow and blue dyes is, of course, always something of a makeshift method, though at times a convenient one. In the case of dyeing cellulose acetate rayon none of the black shades obtainable by means of mixtures of such dyes as those of the SRA Celatene and Dispersol series is so pleasing and fast as the black produced by SRA Black IV. This lack of fastness is, of course, to be expected, since it is almost impossible to select from the limited range of dyes suitable for cellulose acetate rayon three dyes having equal fastness; hence one dye is sure to fade before the others, and thus lead to an alteration of shade. A further important disadvantage of black mixtures is that the total amount of dyes required for but a moderately full black shade is large and the dyeing costs thus become very great. For yarns and fabrics consisting entirely of cellulose acetate rayon, the use of mixtures of dyes for black is not recommended, but in the case of mixed materials containing not less than about 50% of another fiber then it is sometimes convenient to use such mixtures and so avoid treatment of the mixed material with solutions (as required for diazotization and coupling) used with a developed black. In such mixture materials the black color of the rayon contributes to only a proportion of the appearance and may therefore be slightly inferior without greatly affecting the quality of the material as a whole.

Finally we come to the method of dyeing in which the cellulose acetate rayon is first saponified by treatment with a caustic alkali. Since the discovery of the SRA and similar dyes there has been a marked tendency for dyers to turn away from saponification processes. Printers have, of course, long used such processes. But if carefully examined from all points of view, there are no fundamental reasons why cellulose acetate rayon should not be hydrolyzed, provided that it is done satisfactorily. The dyeing of this type of rayon in black shades is a case in point when saponification can often be used to advantage, since the saponified material can then be readily dyed by means of sulphur and other cotton dyes, and also by means of the usual Aniline Black dyeing processes.

It is sometimes stated that on saponification cellulose acetate rayon loses its valuable qualities, particularly that of softness of handle. No doubt this is true if the saponification is continued beyond a certain stage. But if saponification is carried just so far as to give the rayon sufficient affinity for cotton dyes, then it is almost impossible to distinguish between the rayon before and after saponification. Further, the luster of the rayon is only thereby very slightly decreased. On the other hand, saponification of cellulose acetate rayon increases its resistance to hot ironing, which is an important consideration in many instances. So that, on the whole, a dyer should not hesitate to employ a saponification process before dyeing in black shades if it appears advantageous to him.

Usually saponification can be carried out by treating the rayon material with a dilute hot solution of caustic soda. This can be done uniformly in a jig, or it can be carried out in a continuous manner using machines consisting of several boxes, the fabric being saponified in the first box and washed and neutralized in the succeeding boxes. The writer has previously drawn attention to the use of caustic potash instead of soda for the mercerization of cotton materials containing viscose rayon (Jour. Soc. Dyers and Col., 1929, **45**, 171; also this journal, 1929, **18**, 545). Caustic potash has practically no effect on viscose rayon, and it may therefore be clearly seen that caustic potash will be better than caustic soda in the saponification of cellulose acetate rayon. When caustic potash is employed it exerts less solvent action on the superficial layer of cellulose formed on the rayon filaments and also prevents causing the handle of the rayon to become harsh.

In the foregoing summary of the processes suitable for dyeing in black shades materials consisting of cellulose acetate rayon, no mention has been made of the dyeing of mixture materials containing this type of rayon. At the present time cellulose acetate rayon is being used more and more in the construction of cotton, real silk and wool fabrics, so that the black dyeing of these materials is becoming as important as the dyeing of 100% rayon materials. The methods of dyeing mixture materials are various, interesting and sometimes ingenious, but for lack of space they must be dealt with in a separate article.

CIBA PROTEST OVERRULED BY COURT

In a ruling recently handed down by the Customs Court in New York, it is declared that an order signed by the Assistant Secretary of the Treasury, fixing the standards under the dye provisions of paragraphs 27 and 28 of the tariff law, is binding and valid. The Ciba Company's protest, challenging the validity of this order is thus overruled by Judge Brown's written opinion.

Standard Method of Determining Weighting Content Approved

Bureau of Standards Assists Silk Association in Suggesting Tests

A STANDARD method for determining weighting content in silk fabrics, as worked out by W. G. Appel, of the United States Bureau of Standards Department of Commerce, has been approved by the Joint Technical Committee on Weighting and recommended for adoption by the Joint Committee on Weighting. Mr. Appel's report was presented at a meeting of the Joint Technical Committee on Weighting held at the Silk Association of America headquarters.

Determination of a standard method for arriving at the weighting content in silk fabrics has been one of the important subjects upon which the Joint Technical Committee on Weighting have been carrying on research work. Mr. Appel's report is of special significance in view of the fact that it is the first suggested standard method for determining weighting content.

The following resolution was also approved at the meeting to cover glove silk, and will be presented to the Joint Committee on Weighting for adoption:

"Resolved, That a maximum limit of 40 per cent for weighting glove silks be recommended to the Silk Association of America for adoption and addition to the scale of limits known as the Silk Weighting Standards."

Mr. Appel's report, which was presented to the meeting, is in detail as follows:

Standard Method for the Determination of Weighting on Silk

I. General:

1. By "weighting" is meant not only metallic weighting, but all materials other than fibroin present in the finished silk after it has been dried to constant weight in air at 110° C.

2. The amount of weighting is expressed in per cent of the weight of the finished silk after it has been dried, as above.

II. Sampling:

A sample taken for analysis should be representative of the material. It is recommended that a strip measuring from 2 to 4 inches wide be taken all the way across the original cloth from selvage to selvage. The sample should weigh from 1 to 5 grams.

III. Test for Silicate:

A small sample of the silk is ignited, the ash is placed in a platinum crucible and two drops of concentrated hydrofluoric acid are added. If silicate is present, chemical action accompanied by a considerable amount of heat will be noticed.

IV. Tin Phosphate Silicate Weighted Silk.

1. The sample is dried to constant weight in an air oven at 100° C. This is called weight A.

2. The dried sample is soaked in 100 times its weight of distilled water at 65° C. for 20 minutes. It is moved about in the water every few minutes during this time in order to insure thorough penetration of water and extraction of water-soluble materials. It is then rinsed in a fresh portion of distilled water, then in alcohol, and finally in ether, after which it is dried to constant weight, as above (two 25 c.c. portions of alcohol and of ether are usually sufficient). This is called weight B.

$$\frac{\text{Weight A} - \text{Weight B} \times 100}{\text{Weight A}} = \text{Finishing materials in per cent}$$

3. The sample from which "finishing material" has been removed is soaked in 100 times its weight of 2% hydrofluoric acid solution at 65° C. for 20 minutes. It is then rinsed in water and treated in 100 times its weight of 2% sodium carbonate solution at 65° C. for 20 minutes. It is then rinsed in water, in alcohol, and in ether and dried to constant weight as before. This is called weight C.

4. The sample is then ashed and the ash weighted, weight D. This ash should not weigh more than one-tenth of the difference between weight B and weight C. If the silk contains a considerable amount of weighting and treatment given in (3) above should be repeated with fresh solutions before the sample is ashed, in order to obtain a low ash. A few threads of the silk treated along with the sample and ashed will show the operator whether the weighting has been removed in (3) or if the treatment must be repeated.

5.

$$\frac{\text{Weight A} - \text{Weight C} + \text{Weight D} \times 100}{\text{Weight A}} = \text{Weighting in per cent}$$

V. Tin Phosphate Weighted Silk:

1. The procedure for silk which does not contain phosphate is the same as that given in Section 4 above, except that the sample from which finishing material has been removed is soaked in 100 times its weight of 40 per cent hydrochloric acid solution at 55° C. for 20 minutes. This is repeated with a fresh solution. The sample is then rinsed in water and soaked in 100 times its weight of 10% sodium carbonate solution at 55° C. for 20 minutes. It is then rinsed in water and the hydrochloric treatment repeated. The sample is again rinsed in water, then in alcohol and in ether and dried to constant weight, as before. This is called weight C.

VI. Logwood Black Weighted Silk and Silk Weighted with Lead, Zinc, or Aluminum Salts:

1. If phosphate is present, the procedure is the same as that given in Section 4, except that the treatment with hydrofluoric acid is preceded by a treatment with 100 times the weight of the sample of 4% hydrochloric acid solution at 55° C. for 20 minutes, repeated once with a fresh portion of the solution.

2. If silicate is not present, the procedure is the same as that given in Section 5.—*American Silk Journal.*

MATERIALS ARE TESTED BY LIGHT

In many lines of work at the Bureau of Standards light is the principal factor used in the test, says the Commercial Standards Monthly. Some tests are intended to stimulate average daylight conditions, for others only a light of a particular color, or, rather, wave-length is used. In many instances only light or the invisible portion of the spectrum is used—the ultra-violet and the infra-red—but because the weather is such an uncertain element that the scientist cannot depend upon the sun as a reliable source of energy for his tests. Quick, reliable results under conditions that can be duplicated and which are comparable with solar radiation are necessary.

A careful spectral analysis was made of the light emitted by the sun and compared with artificial light sources, and for many tests, especially in the study of the fading of fabrics, paper and inks, the carbon-arc lamp is used because its light has been found the closest approach to that of average sunlight. The time of exposure of samples to such light is extended as much as seems necessary. In some cases, as when testing paints and varnishes, a water spray is used to bring in the effect of rainfall. Most of the work mentioned is in the experimental stage so that just how particular kinds of material will stand up remains to be seen.

Everyone knows how difficult it is to obtain an exact color match for cloth, and it is quite a problem even for the manufacturer to duplicate any desired shade, for some interesting work being done by the bureau's textile division may solve the difficulty. A spectro-photograph is taken of a sample, and by means of certain prominent lines or bands in the spectrum the dye for that material is definitely identified and can be exactly duplicated. Other questions entering into the dyeing of a fabric must be considered, but these should be within control of the manufacturer. The study is under way at the bureau.

Incandescent lamps are tested to insure that the lamps supplied the Government, under contract, comply with the specification for candlepower and life. Lamps of an entirely different type are calibrated for manufacturers for use in pyrometers to enable them to keep foundry operations in the working of metals well within the critical temperature ranges required.

By the rotation of a beam of plane polarized light passing through sugar solutions the percentage of sugar is obtained. By this method the bureau's sugar laboratory checks the laboratories of the Custom Service, and the duty collected by the Government on imports is based on the results of these tests.

For some kinds of work monochromatic light, or light dominant in one particular color, is used, more particularly to ascertain differences in dimension between two objects or to determine those minute but important changes which occur with change in temperature or when material is in a stressed condition. By interference of light waves a pattern of light and dark bands is disclosed, the distance from one light or dark band to another light or dark band represents one-half wave-length. This method is used to check the master gauge blocks for manufacturers, the accuracy of the depth of blood counting chambers used by physicians, dental amalgams, enamels for metal ware, glazes for porcelains, etc.

These are only some of the ways in which light is used to test materials at the bureau, but there are many others.

T. C. C. A. WINTER CARD ANNOUNCED

A special Winter Card featuring eighteen shades has just been issued to members of the Textile Color Card Association, it was announced by Margaret Hayden Rorke, managing director. The card portrays the color highlights of the recent Paris openings, as well as other outstanding color developments for the 1929-30 winter season.

Five purple shades are featured on the new card, ranging from an animated tone for evening wear to a rich dark shade almost black. These are appropriately named Magenta Dahlia, Royal Dahlia, Violet Dahlia, Purple Dahlia and Iris Noir.

Greens are accorded a place of importance on the card and appear in four different casts. Gala Green is a light vivacious tone with a yellowish tint, intended for evening wear; Emeraude is a bright emerald green; Antibes Green is a medium shade with a bluish cast, while Carafe Green is a very dark bottle green.

Two shades of brown are significant. Mauve Brown, as its name implies, is a dark brown with a subtle mauvish undertone. Cocoa Crème is a warmer medium brown.

Blues are represented in two tonalities. Bleu de Cannes is a very smart darker tone with a purplish cast, while Blue Eyes, for evening wear, is a deep sky blue with a suggestion of violet.

Two red shades are a feature of the card. Grenadine is a rich mellowed red, while Rouge Basque is the lively light orange red one associates with the picturesque Basque country. Rose Laque is a new rust shade with a decided rose overtone.

The card will be available to the trade shortly.

JAPANESE DYESTUFFS COMPANY EXPANDS

Assistant Trade Commissioner Howard B. Titus at Tokio reports that it is generally believed that the Mitsubishi Dyestuffs Company is enlarging its factory and will shortly commence manufacturing Malachite Green on a larger scale than before. It is stated that the manufacturing process has been perfected by experts of the Government Industrial Experiment Station at Fukuyama, and that the feature of their improvement is a very low priced raw material which can be utilized in the production of this green dye. It is anticipated that the price of this dye will be 20% less than at present. The process of manufacture will not be patented. It is thought that this company will be able to produce sufficient Malachite Green to fulfill all domestic demands.

Soaps

Part II

Recovering Glycerine—Soap Manufacture—The Oils and Fats Used—Purchase of Soaps—Wool Scouring—Washing Cotton and Silk

By J. M. HOOD

[*Editorial Note—This article was written especially for dyers, finishers and salesmen who have not had the benefit of a technical education but who would like to increase their knowledge in regard to the chemical nature of soaps and their uses in the textile industry.*]

IN actual practice when making a soap from a fat usually an excess of caustic soda is employed for boiling. When the boiling has been completed we have in the soap tanks a solution of soap and glycerine and excess caustic soda. In order to get rid of the excess caustic and the glycerine, sodium chloride (table salt) is shoveled into the soap tank and dissolved. When enough salt has been added the salt will split the contents of the soap tank into two layers, the top layer being melted soap and the bottom a solution of salt, glycerine and the excess caustic. This operation the soap maker calls salting out and depends upon the fact that soap is insoluble in a strong salt solution. The lower water layer of glycerine, excess caustic soda and salt is now drawn off, leaving the soap behind. The solution of glycerine, excess caustic soda and salt now goes to a recovering plant where the valuable product glycerine is recovered, as is also the salt and caustic. Sometimes these materials are not recovered and go to the sewer.

The liquid soap remaining in the tank is drawn off, water being added if need be to give the soap content wanted in the finished soap. In some cases after the glycerine layer has been drawn off the soap is given another boil with more caustic soda and salted out again, and this operation of boiling up and salting out may be repeated again when an exceptionally pure soap free from excess caustic and unsaponified fat is wanted.

The accompanying table is an interesting one, taken from "Martin's—The Modern Soap and Detergent Industry," and may be considered a condensed story of soap making. Do not become alarmed at the bigness of the names or the size of the formulas or the number of carbon and hydrogen atoms in each one. The points to get are:

First.—A series of soaps exist containing from one carbon atom up to twenty-nine carbon atoms. See column 1 and 2 on the table. The last part of the formula for a soap is always pictured as COONa, the only difference in the chemical formula being in the number of carbon atoms in the chain or row of carbon atoms preceding the COONa and in the number of hydrogen atoms attached to the carbons.

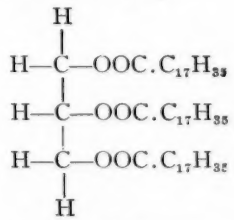
Second.—A series of fatty acids exist from which soaps can be made. See columns 3, 4, 5 and 6. The picture formula of a fatty acid always ends in COOH, the only difference between the fatty acids being due to the number of carbon atoms in the formula preceding the COOH and the number of hydrogen atoms attached to the carbons.

Third.—A series of fats or glycerides of the fatty acids exist from which soaps can be made by boiling with alkalies. In the fats one molecule of glycerine is always combined with three molecules of fatty acids, fats differing from one another depending on the number of carbon atoms in the chain. See columns 7, 8, 9, 10, 11, 12.

Taking the fat stearin as a typical example—if we were to make a sodium soap from each molecular weight of stearin we would require three molecular weights of caustic soda (NaOH). That is, 890 pounds of stearin would require 120 pounds of NaOH. This figured as percentage NaOH absorbed by 890 pounds of stearin would be $\frac{120}{890}\ 100 = 13.48\%$ or the figure obtained under column 10 for stearin.

Likewise column 11 shows us that for every 890 pounds stearin used in soap making, 92 pounds of glycerine are set free. This figured to per cent on the weight of the original glyceride or fat gives us in column 11: $\frac{96}{890}\ (100) = 10.34\%$ glycerine set free.

Thus, the formula for stearin is $C_3H_5(O.C_{17}H_{35}CO)_3$, or it may be expressed pictorially as:



or by addition we find that it contains 57 carbons, 110 hydrogen and 6 oxygen atoms. The molecular weight is thus:

$$54 \times 12 + 40 \times 1 + 6 \times 16 = 890.$$

(12 equals atomic weight of carbon, 1 equals atomic

TABLE OF PRINCIPAL SODIUM SOAPS

Soaps of Saturated Fatty Acids of the Acetic Series (General Formula CnH2m —1.COOH)

Sodium Soap (1)	Formula (2)	Corresponding Fatty Acid (3)	Formula (4)	Molecular Weight (5)	Per Cent NaOH Absorbed (6)	Corresponding Glyceride (7)	Formula (8)	Molecular Weight (9)	% NaOH Absorbed (10)	% Glycerol Set Free (11)	% Fatty Acid Set Free (12)
Sod. Formuate	$H.COONa$	Formic	$N.COOH$	46	86.96	Formin	$C_3H_5(O.HCO)_3$	176	68.19	52.28	78.41
Sod. Acetate	$CH_3.COONa$	Acetic	$CH_3.COOH$	60	66.67	Acetin	$C_3H_5(O.CH_3CO)_3$	218	55.04	42.20	82.56
Sod. Propionate	$C_2H_5.COONa$	Propionic	$C_2H_5.COOH$	74	54.06	Propionin	$C_3H_5(O.C_2H_5CO)_3$	260	46.15	35.38	86.14
Sod. Butyrate	$C_3H_7.COONa$	Butyric	$C_3H_7.COOH$	88	45.45	Butyrin	$C_3H_5(O.C_3H_7CO)_3$	302	39.74	30.47	87.42
Sod. Valerate	$C_4H_9.COONa$	Valeric	$C_4H_9.COOH$	102	39.22	Valerin	$C_3H_5(O.C_4H_9CO)_3$	344	34.88	26.74	88.94
Sod. Caproate	$C_5H_{11}.COONa$	Caproic	$C_5H_{11}.COOH$	116	34.48	Caproin	$C_3H_5(O.C_5H_{11}CO)_3$	386	31.09	23.83	90.16
Sod .Cenanthylic	$C_6H_{13}.COONa$	Cenanthylic	$C_6H_{13}.COOH$	130	30.77	Cenanthylin	$C_3H_5(O.C_6H_{13}CO)_3$	428	28.04	21.50	91.14
Sod. Caprylate	$C_7H_{15}.COONa$	Caprylic	$C_7H_{15}.COOH$	144	27.77	Caprylin	$C_3H_5(O.C_7H_{15}CO)_3$	470	25.54	19.57	91.91
Sod. Perlargonate	$C_8H_{17}.COONa$	Perlargonic	$C_8H_{17}.COOH$	158	25.31	Perlargonin	$C_3H_5(O.C_8H_{17}CO)_3$	512	23.44	17.97	92.58
Sod. Caprate	$C_9H_{19}.COONa$	Capric	$C_9H_{19}.COOH$	172	23.26	Caprin	$C_3H_5(O.C_9H_{19}CO)_3$	554	21.66	16.61	93.13
Sod. Undecylate	$C_{10}H_{21}.COONa$	Undecylic	$C_{10}H_{21}.COOH$	186	21.51	Undecylin	$C_3H_5(O.C_{10}H_{21}CO)_3$	596	20.14	15.44	93.63
Sod. Laurate (*)	$C_{11}H_{23}.COONa$	Lauric	$C_{11}H_{23}.COOH$	200	23.00	Laurin	$C_3H_5(O.C_{11}H_{23}CO)_3$	638	18.81	14.42	94.06
Sod. Tridecylate	$C_{12}H_{25}.COONa$	Tridecylic	$C_{12}H_{25}.COOH$	214	18.27	Tridecylin	$C_3H_5(O.C_{12}H_{25}CO)_3$	680	17.65	13.53	94.41
Sod. Myristate (*)	$C_{13}H_{27}.COONa$	Myristic	$C_{13}H_{27}.COOH$	228	17.55	Myristin	$C_3H_5(O.C_{13}H_{27}CO)_3$	722	16.63	12.75	94.75
Sod. Pentadecatoate	$C_{14}H_{29}.COONa$	Pentadecatoic	$C_{14}H_{29}.COOH$	242	16.53	Pentadecatoin	$C_3H_5(O.C_{14}H_{29}CO)_3$	764	15.70	12.04	95.11
Sod. Palmitate (*)	$C_{15}H_{31}.COONa$	Palmitic	$C_{15}H_{31}.COOH$	256	15.63	Palmitin	$C_3H_5(O.C_{15}H_{31}CO)_3$	806	14.89	11.41	95.30
Sod. Daturate	$C_{16}H_{33}.COONa$	Daturic	$C_{16}H_{33}.COOH$	270	14.81	Daturin	$C_3H_5(O.C_{16}H_{33}CO)_3$	848	14.16	10.85	95.52
Sod. Stearate (*)	$C_{17}H_{35}.COONa$	Stearic	$C_{17}H_{35}.COOH$	284	14.09	Stearin	$C_3H_5(O.C_{17}H_{35}CO)_3$	890	13.48	10.34	95.72
Sod. Nondecylate	$C_{18}H_{37}.COONa$	Nondecylic	$C_{18}H_{37}.COOH$	298	13.43	Nondecylin	$C_3H_5(O.C_{18}H_{37}CO)_3$	932	12.87	9.87	95.92
Sod. Arachidate	$C_{19}H_{39}.COONa$	Arachidic	$C_{19}H_{39}.COOH$	312	12.82	Arachidin	$C_3H_5(O.C_{19}H_{39}CO)_3$	974	12.32	9.44	96.10
Sod. Medullate	$C_{20}H_{41}.COONa$	Medullic	$C_{20}H_{41}.COOH$	326	12.28	Medullin	$C_3H_5(O.C_{20}H_{41}CO)_3$	1,016	11.81	9.06	96.27
Sod. Behenate	$C_{21}H_{43}.COONa$	Behenic	$C_{21}H_{43}.COOH$	340	11.77	Behenin	$C_3H_5(O.C_{21}H_{43}CO)_3$	1,058	11.34	8.69	96.40
Sod. Carnaubate	$C_{23}H_{47}.COONa$	Carnaubic	$C_{23}H_{47}.COOH$	368	10.87	.....	.....	.....	.....	.....	.....
Sod. Hyaenate	$C_{24}H_{49}.COONa$	Hyaenic	$C_{24}H_{49}.COOH$	382	10.47	.....	.....	.....	.....	.....	.....
Sod. Cerotate	$C_{26}H_{53}.COONa$	Cerotic	$C_{26}H_{53}.COOH$	410	9.76	.....	.....	.....	.....	.....	.....
Sod. Melissate	$C_{29}H_{59}.COONa$	Melissic	$C_{29}H_{59}.COOH$	452	8.85	.....	.....	.....	.....	.....	.....

Soaps of the Unsaturated Fatty Acids of the Ricinoleic Series (General Formula CmH2n —2OH.COOH)

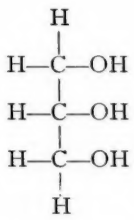
Sod. Ricinoleate (*)	$C_{17}H_{32}.COONa$	Recinoleic	$C_{17}H_{32}.OH.COOH$	289	13.43	Recinolein	$C_3H_5(O.C_{17}H_{32}.OH.CO)_3$	932	12.87	9.87	95.92

*Oleic acid ($C_{17}H_{35}.COOH$), left from table by mistake, contains 2 hydrogen atoms less than stearic acid, and finds extensive use in soap making. It is an unsaturated acid and is sold as red oil or Elaine oil.

(*Most important ones.)

weight of hydrogen, 16 equals atomic weight of oxygen.)

Likewise, the formula for glycerine is C_3H_5OH pictured:



or by addition we find that it contains 3 carbon, 8 hydrogen and 3 oxygen atoms. The molecular weight is:

$$3 \times 12 + 8 \times 1 + 3 \times 16 = 92.$$

Thus, we see that one molecular weight of stearin (fat) (glyceride of stearic acid) will give one molecular weight of glycerine; 890 parts of stearin will give 92 parts of glycerin or calculating as pounds, 890 pounds of stearin (fat) (glyceride of stearic acid) will give 92 pounds of glycerine. Thus, the percentage of glycerine obtainable is 10.34:

$$\frac{92}{890} \times 100 = 10.34$$

Likewise, column 12 shows us that for every 890 pounds of stearin used in soap making, 284 pounds of stearic fatty acids are produced, or 95.72% of the original weight of the glyceride.

The manufacture of soap from a pure fat or glyceride has been discussed, using the glyceride of stearic acid as an example.

In the manufacture of soaps from commercial fatty acids and fats (glycerides) we do not deal with pure glycerides such as stearin (glyceride of stearic acid), which we have used as an example of a soap formula.

The fats used in soap making are mixtures of various glycerides. However, a good many of the glycerides exist in fats in very small percentages, and in the last table the most common fatty acids which occur in glycerides or in commercial fatty acids have been marked (*).

The following oils and fats are used in soap making and a list of the principal or predominating glycerides occurring in each is given below:

Tallow—Composed of:
- Tristearin (glyceride of stearic acid)
- Tripalmitin (glyceride of palmitic acid)
- Triolein (40% to 46%) (glyceride of oleic acid)

Cocoanut Oil—Composed of:
- Glyceride of Lauric acid
- Glyceride of Myristic acid
- Glyceride of Palmitic acid.

This fat can be saponified in the cold because of the presence of fatty acids of low carbon content (Lauric and Myristic) which saponify more easily than higher carbon content fats similar to stearin).

Olive Oil is composed of:
- 70% Triolein
- 28% Tripalmitin and stearin.

Castor Oil:
- Glyceride of Ricinoleic acid
- Isoricinoleic acid
- Dihydroxystearic acid
- And little Tristearin.

Castor oil is composed mainly of the glycerides of unsaturated fatty acids.

Palm Oil—Composed of:
- Tripalmitin
- Triolein
- And free acids.

Purchase of Soaps

A good many mills pay excessive prices for their soaps, often paying a high price for water. This is poor business for the company purchasing the soap, but profitable for the company selling.

Many soaps on the market contain, along with the expensive soap, a large proportion of other chemicals, which cost much less than soap, but are sold at soap prices. If a concern is a large consumer of soaps it should have the products it purchases analyzed to see just what they are buying. Do not buy 88% filler worth 1½ cents a pound for 88% soap worth 10 cents a pound.

The price paid for soaps sold as 88% soaps and containing 12% moisture may be considered the standard upon which to judge the price of soaps containing more water. The following prices are typical for soaps now on the market:

Flaked Soaps:
- 88% Tallow soap chipped—10½ cents per pound.
- 88% Palm Oil soap—10¼ cents per pound.
- 88% Olive Oil—12 cents per pound.
- 88% Red Oil flake—12 cents per pound.

Solid Soaps:
- Palm Oil—pure, 65 to 68%, anlydrous, dry, 8 cents.
- Olive Oil—pure, 65 to 68% anlydrous, dry, 9 cents.
- Red Oil—pure, 42 to 44% anlydrous, dry, 9 cents.

Potash Soaps:
- Olive fig.—42 to 44%, 8 cents.
- Red Oil fig.—42 to 44%, 7⅝ cents.

Soaps find extensive use in the textile industry, and an outline of their use in the various branches of the industry follows:

Wool.—Soap is used for the scouring of raw wool as it comes from the back of the sheep. Raw or unscoured wool contains varying amounts of dirt, grease and dried perspiration, the quantity depending upon the type of

sheep and the locality in which they are raised. This dirt and grease prevents the proper carding, combing, drawing, spinning, weaving and finishing of the wool and must be removed by wool scouring. One hundred pounds of wool as clipped from the sheep may give but 50 pounds of clean wool after the scouring operation. The scouring is done by the washing of wool in warm dilute soap solutions to which other assistants, such as ammonia, or sodium carbonate (soda ash) or potassium carbonate are used. Caustic soda and caustic potash have been used in wool scouring, but these materials should not be used except under strict supervision and by men who realize the damage that may result by misuse. Weak solutions of caustic soda or potash will dissolve wool at the boil, and this fact should always be borne in mind when wool and these chemicals are within reach of one another.

For wool scouring pure soaps must be used. They must not contain free caustic soda or potash, except it be known to the wool scourer, no fat which has not been converted into soap, no mineral oil, no rosin and must not leave an odor in the scoured wool.

The scouring of wool takes place usually at temperatures below 130° F., as above that temperature the soapy liquors begin to attack and injure the wool fiber.

Potash soaps are known as soft soaps and are held superior to soda soaps, known as hard soaps, for wool scouring because they are considered to be more soluble and easier to wash out than soda soaps. Just now this point is being disputed and the Procter & Gamble Company has submitted to the trade circulars which tend to show that soda soaps of certain fats are as soluble as the potash soaps. Soda soaps are much cheaper, and laboratory tests made using soda soap in place of the more expensive potash give the same results. A soda red oil soap (oleic acid) was used against a similar potash red oil soap.

Potash olive oil soap is an ideal soap for wool scouring, but is expensive, and in its place potash red oil soaps are often used. For the lower grades of wool hard soaps similar to soda tallow soaps are used along with soda ash.

The amount of soap and other materials to be used in scouring wool is usually left to the judgment of the operator of the scouring machine. If he does not use enough soap his wool comes through dirty and has to be rewashed, while if he uses too much soap he is bothered by the wool becoming slippery and too much foaming.

The following is taken from "Cloth Finishing," by J. Schofield: "The concentrations of soap and other material used vary greatly, but the following data represent an average case:

Bowl No. 1—1,800 gallons soap (0.75%), alkali, 0.25%.
Bowl No. 2—1,500 gallons soap (0.50%), alkali, 0.10%.
Bowl No. 3—1,000 gallons soap (0.25%), alkali, 0.10%.
Bowl No. 4—Water only.

Or figuring over to pounds of soap (50% water) and pounds soda ash per 100 gallons, it would be:

Bowl No. 1—12 pounds potash soap—50% water and 2 pounds soda ash per 100 gallons.

Bowl No. 2—8 pounds potash soap—50% water and 1 pound soda ash per 100 gallons.

Bowl No. 3—2 pounds potash soap—50% water and 1 pound soda ash per 100 gallons."

The following information was abstracted from Mathews' Bleaching and Related Processes: "Fourteen lots of various grades of greasy wool were scoured with the following results:

Total weight of greasy wool	11,000 lbs.
Total yield of clean wool	7,476 lbs.
Per cent yield of clean wool	68%
Actual consumption of soap	128 lbs.
Per cent soap used on weight of greasy wool	1.1%
Actual consumption of soda ash	362 lbs.
Per cent consumption of soda ash on weight of greasy wool	3.3%
Time required for scouring	13.23 hrs."

Mathews also states that for ordinary grades of wool the following soap and soda ash is used:

2 lbs.—10 lbs.—soap for every 100 gallons of water.
10 lbs.—20 lbs.—soda ash for every 100 gallons of water.

In some plants excellent results will be obtained scouring with 4% soda ash and 2% potash red oil soap (50% water) on the weight of the wool, while in others the reverse percentages of soap and soda ash may be used to obtain similar results. From a cost point of view, scouring with high soda ash and low soap is the cheaper method. Soda ash costs about 1½ cents a pound; red oil potash soap containing 50% water about 8 cents a pound. Thus, 100 pounds wool scoured with 4 pounds soda ash and 2 pounds 50% potash red oil soap (50% water) will cost 22 cents. One hundred pounds wool scoured with 4 pounds potash red oil soap (50% water) and 2 pounds soda ash will cost 35 cents.

After the wool has been scoured of its dirt and oil, it is necessary to re-oil the wool to improve its spinning qualities, and the amount of oil thus put back into the wool may amount to 10% on the weight of the wool. The best oil used for this purpose is olive oil, but sometimes inferior oils are used and not infrequently mineral oils. The piece of cloth as woven contains this added oil along with grease and dirt picked up from the floors and machinery. It is now necessary to remove this dirt and oil, and the process will vary depending upon the types of oil to be scoured out. Soap plays an important part in this operation, and if soap is not added directly to the scouring bath it is formed directly in the bath from the oil used for oiling the wool reacting with soda ash to make the soap. Very often soap and soda ash solutions are used in the operations as well as ammonia. Special soaps for the process are on the market, but in nine times out of ten are better left alone. Usually a high price is paid for an abundant supply of water, and in but few

cases have any advantage over the use of old-fashioned soap and soda ash. The beneficial results to be obtained from most of them are open to dispute, soap and soda ash when properly used doing very good work.

As in the case of the scouring of raw wool, the temperature is usually kept below 130° F., so as not to injure the wool fiber, and caustic soda or caustic potash must not be used except under strict supervision. The amount of soap and other materials to be used depends in a large degree upon the judgment of the operator. Too little soap and added chemicals means poor work, and too much means trouble with foaming and the danger of injury to the fiber or of felting. For this type of scouring soda red oil soap seems to be the one most used, although for cheaper work cheaper soaps can be used. For an average scour not over one ounce of an 88% soda red oil soap, and half an ounce of soda ash per gallon would have to be used. Depending upon the materials the amounts of soap and soda used would be increased or decreased.

Soaps are also used in the fulling or milling of textile cloth. A fulling machine is so constructed that the cloth saturated with soap is subjected to a pressure which causes the fabric to shrink either in length or breadth or in both directions. At the same time the fibers become more or less felted together, the amount being dependent upon the type of cloth being fulled. In the finishing of woolen clothes, fulling plays a major part and may be carried on for several hours, but in the case of worsteds is a minor operation and takes place for only a few minutes, if at all. Fulling soaps must be easily soluble in water and contain no free caustic. The best soaps for fulling are olive oil soft soaps. They are, however, expensive, and in their place are usually used soda palm oil or tallow soaps. Both yield a heavy bodied soap, but are not as soluble as potash olive oil soap. Fulling is carried on cold or moderately warm, and the strength of the soap solution used varies anywhere from a few ounces to a pound per gallon of a 70% palm oil soap. A soap of the latter concentration will be a heavy jell.

Cotton.—Cotton is not as dirty and oily as wool, and soap is used mostly for the scouring out of yarn or cloth in the cleansing operation preceding the dyeing, during and after the dyeing, and in the printing of cotton.

Cotton cloth is prepared in several ways for dyeing or for selling as white or bleached goods. The cloth may be put into large iron tanks called kiers, holding five tons of cloth, and have circulated through it at temperatures ranging from the boiling point of water, 212° F. up to 230° F., a solution of caustic soda, usually about 3° Tw. Sometimes soap is added to the kier liquor, and this operation of kier boiling, if property carried out, yields a piece of cloth capable of being bleached to a good and lasting white and being very water absorbent. This operation removes most of the cotton waxes and fats from the cotton and is quite a severe treatment.

Oftentimes kier boiling is not necessary and the cotton cloth receives a scouring out which softens the waxes, but does not remove them. In this operation various formulas for the scouring bath are used, and there are many specially prepared, often high priced, scouring soaps for which great claims are made. Where soft water is available a very good scour can be had by the use of a good potash olive oil soap, or a potash red oil soap, or a soda red oil soap using not over 1 ounce of 50% soap per gallon and maintaining the soaping temperature at 170° to 180° F., rinsing in warm water. The amount of soap used will have to be varied, depending somewhat upon the type of machine used, the amount of foam produced, etc. Several chemists, working independent of one another, after making a series of tests with the soaps mentioned above against various types of the solvent soaps now on the market and costing much more money, came to the same conclusion: "Working at a temperature of 170° to 180° F. and using a good potash olive oil foots, or potash red oil or soda red oil soap, you obtain just as good if not a better scour than with the solvent soaps." A little soda ash (1 ounce gallon) or caustic soda (½ ounce gallon) can be used but must be washed out before the cloth is dried or tendering of the cloth may result.

Soaps are also used to clean cotton cloth after it has been printed. In this operation the soap helps to remove the gums and starch used in the printing pastes, removes loose color and brightens up the colors. A good grade of soap and one that will not leave an odor in the goods must be used. Tallow or palm oil soaps are used but potash or soda olive oil soaps are the best.

Soap and the Silk Industry

Soap is used in the silk industry to degum the silk, and during aftertreatment of the goods for dyeing.

Raw silk consists of two portions, the outer called sericin, gum, or glue, and which constitutes 20 to 30% of the raw silk, and the inner called fibroin, which is the true silk fiber.

In order to remove this outer layer of gum and thereby obtain the full luster and softness of the silk, the silk is boiled with soap solution. The strength of the soap solution used and the length of time the silk is in the soap bath varies, depending upon the quality of the silk and the finish desired. The gum is not always completely removed.

The best soaps used for degumming silk are soda and potash, olive oil or peanut oil soaps, and red oil soaps. Usually ½ to 1 ounce of soap (50%) is used per gallon.

The soap liquor used in boiling off the gum from the raw silk is known as boiled-off liquor, and is used as an addition to the dye bath.

The following are typical formulas which are used in the manufacture of textile soaps:

Tallow Soda Soap: Not salted, contains glycerine from the fat and a little free caustic soda (NaOH):

1150 lbs. tallow
50 gal. caustic soda (3.48 lbs. dry caustic per gal.)
3300 lbs. water

5000 lbs. soap

This gives a 25% almost neutral soap used for the scouring of cotton cloth and yarn.

Soda Red Oil Soap—Salted Out

2160 lbs. red oil (oleic acid)
150 gal. caustic soda (3.48 lbs. solid caustic per gal.)
200 lbs. water
300 lbs. salt

This represents a soap made from fatty acid and is used for scouring woolen and worsted cloth.

Potash Red Oil Soap

3150 lbs. red oil (oleic acid)
750 lbs. KOH solid
300 lbs. K_2CO_3
2600 lbs. water

6800 lbs. soap.

This represents a soft soap made from potash and used in wool scouring. It contains a small percentage of free carbonate of potash.

Dyeing Possibilities of Acetate Rayon, Alone and in Mixtures

The Properties of Acetate Rayon—Its Use Alone—Dyestuffs—Rayon in Mixed Weaves—Dyes for Various Combinations

By GEORG RUDOLPH

(*Translated from Die Kunst-Seide*, 11, 261 (1929))

WITH a product such as acetate rayon, it is not surprising that often there appear in many journals articles regarding it, which are read with interest, although one often comes upon statements of facts already familiar to him. Any work of the sort involves citation of previously published points of view, for further clearing up of the point involved, or for confirmation or correction. The author deals with the subject of the general usefulness of acetate rayon, and with the most important dyeing procedures, with special reference to the newer dyestuffs proposed for use with it.

Artificial silk is, of course, a new textile fiber, so that it is not a question of its being a substitute for natural silk, cotton or linen. The new fiber has found entirely new fields of usefulness opened to it, since its striking beauty attracted much attention and led to its ever increasing use. Though the use of cotton, for example, has considerably diminished because of the increasing use of artificial silk stockings, yet the use of natural silk has not diminished, but has even increased. Before the year 1914 altogether simpler garments were being worn, and it is true that, for years before, womankind had been utilizing more and more lustrous fabrics, in order to increase their attractiveness to others. And rightly so, for anyone who finds pleasure in luster and beauty, who enjoys the sight of a gaily-painted butterfly or a well-kept garden, appreciates the beauty of life incomparably more than the cynic who stands apart and feels no response in himself to what is beautiful.

When acetate rayon, not many years ago, had passed through the experimental stage, there were, as always with anything new, doubters who questioned whether it could continue to exist, or whether it would ultimately prove to be of little importance after all. Many were skeptical, but the course of events convinced them of the true value of the new product.

The fine fiber of acetate rayon is soft, elastic, smooth, and uniform. Its luster is dull, its feel and draping characteristics are like those of natural silk. It also resists the effect of water better than do the other fibers. A further point in its favor is that it is completely resistant to vermin, moths, etc., that it keeps the body warm in winter and cool in summer, when used in underclothing. These are the reasons for its rapid climb into favor. Its behavior toward the processes of dyeing is of unusual interest and value. It is possible to attain with it effects in mixed weaves which literally never could have been imagined. We may suppose that this fact was what gave the weaving industry the incentive to those further creations, which have awakened such universal interest. We take up the most significant dyeing methods here in order to point out the way to further achievements, mentioning only those dyestuffs or groups of dyestuffs which are specially suitable for acetate rayon alone or for mixed weaves containing it, and which are particularly useful for producing two-color or polychrome effects.

Acetate Rayon Alone

1. *The Cellite Fast Dyestuffs.*—Of these we have, as older products, yellow, orange, a handsome red, two violets, and a pure blue. Recently there have come upon the

(*Continued on page* 685)

Proceedings of the American Association of Textile Chemists and Colorists

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The Nomination Committee submit the following nominations for officers for 1930:

President—Dr. E. H. Killheffer, President, Newport Chemical Company, Passaic, N. J.

Vice-Presidents—Walter S. Williams, Purchasing Agent and Chemical Engineer, Mount Hope Finishing Company, North Dighton, Mass., and P. J. Wood, Vice-President, Oriental Silk Printing Company, Paterson, N. J.

Treasurer—Harry R. Davies, President, Dyestuff Corporation of America, 281 Franklin Street, Boston, Mass.

Secretary—Alex. Morrison, Assistant Chemist, American Woolen Company, Andover, Mass.

Councilors at Large to 1933—George A. Moran, Chief Chemist, Pacific Mills, Lawrence, Mass.; Hugh Christison, Chief Chemist, Arlington Mills, Lawrence, Mass.

The Committee wish to call attention of the members to the latter part of Paragraph 3 in Article VIII of the Constitution, on page 60 of the 1928 Year Book, which reads:

"Counter nominations for any office, signed by at least ten members, may be filed with the Secretary not later than November 1st."

ALEX. MORRISON, *Secretary.*
Chairman, Nomination Committee.

Joint Meeting of A. A. T. C. C. and S. T. A. in Charlotte

The Piedmont Section of the American Association of Textile Chemists and Colorists and the Dyers, Finishers, and Bleachers Division of the Southern Textile Association will hold a joint meeting in Charlotte, N. C., on Saturday, October 26, 1929.

Both of these associations usually have a very large attendance, and, with the excellent program now in preparation, it is expected that this will be the largest meeting of textile chemists and dyers ever held in the South, if not the entire United States.

The program is now in the hands of the local chairmen of the respective associations, Professor Mullin and Paul Haddock, and will be announced at an early date.

Applicants for Membership

Active Membership

Gladue, Rodolphe A., chief chemist, Herrick & Voigt Chemical Corporation, 141 East Fifth Street, Bayonne, N. J. Sponsors: P. F. Kingsbury and Thomas R. Harris.

Morgan, Edward W., president, Morgan Dyeing & Bleaching Company, Rochelle, Ind. Sponsors: C. W. Wille and A. T. Brainerd.

Pozzi, Charles M., dyer and bleacher, Arnold Print Works, North Adams, Mass. Sponsors: Albert S. Jones and Denis de Goenoz.

Sefton, Archibald K., chief chemist, Dyestuff Manufacturing Control Laboratory, Du Pont Dyeworks, Deepwater Point, N. J. Sponsors: Charles A. Seibert and H. G. Fish.

Turner, J. A., salesman, E. I. du Pont de Nemours & Co., 1114 Union Trust Building, Chicago, Ill. Sponsors: Fred E. Beecher and C. W. Wille.

Wood, Robert P., foreman, color shop, Slater Company, Inc., Webster, Mass. Sponsors: Frank C. Hewitt and Harold C. Bloxham.

Junior Membership

Burns, William M., salesman, United Indigo & Chemical Company, 220 West Kenzie Street, Chicago, Ill. Sponsors: Hugh F. McKenna and Henry Waffenschmidt.

Hopkins, G. Elberne, chemical engineer, Bigelow Hartford Carpet Company, Thompsonville, Conn. Sponsors: L. A. Olney and Harold C. Chapin.

Kingman, A. E., chemist, Morgan Dyeing & Bleaching Company, Rochelle, Ill. Sponsors: N. A. Ruston and Fred E. Beecher.

Spink, Douglas G., application chemist and assistant dyer, Arnold Print Works, North Adams, Mass. Sponsors: Albert S. Jones and Denis de Goenoz.

NOTICE

The Secretary would like the present addresses of the members whose names and last known addresses appear below:

Bloxham, Harold C., 88 Linwood Avenue, Pawtucket, R. I.

Frazee, M. D., Box 274, Nashua, N. H.

Green, L. F., 232 West First Street, Chattanooga, Tenn.

Photis, Christos J., 466 West Twenty-third Street, New York, N. Y.

Schoffstall, Chas. W., 821 Forest Avenue, Evanston, Ill.

Williams, S. H., General Dyestuff Corporation, Chattanooga, Tenn.

Reports Bound

The Research Committee has had bound the three reports of the Sub-Committee on Light Fastness, and these bound copies can be procured from the Secretary at 60 cents a copy.

ANNOUNCING

the

Annual Meeting, American Association of Textile Chemists and Colorists

Bellevue Stratford, Philadelphia

December 6th and 7th, 1929

(*Continued from page* 682)

market Cellite Fast Red BB, a lively bluish-red of good fastness to light, and Cellite Fast Brown R, a yellow-brown. All these dyestuffs dissolve completely in hot water. According to the depth of shade, dyeing is carried out with addition of 20% to 50% of Glauber salt calcined, or ammonium chloride, while for lighter shades the goods are entered at a low temperature and dyed at 60° to 70° C. An exception to this method is required by Cellite Fast Yellow GGN, which gives considerably weaker shades if dyed only with Glauber salt, but used with addition of 2% to 5% of acetic acid it yields dyeings of full strength and exhausts well. If dyeing with the dyestuff alone, the amount of acetic acid mentioned is used and only up to 30% of Glauber salt. It is interesting to observe that the other Cellite Fast dyestuffs can also be dyed from an acid bath. Such a bath is used, if it is a question of mixture with Cellite Fast Yellow GGN. The new Cellite Brown R is best not dyed with Glauber salt, as it does not take well on the fiber with that assistant; it is dyed with 20% to 50% of ammonium chloride or common salt. If Cellite Brown R is to be mixed with the other Cellite Fast colors, ammonium chloride is used as the assistant, or, if Cellite Fast Yellow GGN is a component of the mixture, about 2% of acetic acid also. It is possible, therefore, to mix the Cellite Fast colors with each other without special precautions needing to be taken; and innumerable tones can thus be produced with them.

2. *The Cellitone and Cellitone Fast Colors.*—A recent product in this group is Cellitone Fast Blue Green B paste, which gives a very handsome, bright tone similar to a Patent Blue, and of decidedly good fastness to light. Of interest also are the new Cellitone Fast Marine Blue B and GT pastes; the first gives a bright, the second a more covered, marine blue. The fastness to light of B is very good, of GT good. Cellitone Fast Black B and G pastes are new, full blacks, of which B is faster to light than G, both being good to very good. Apparently an effort is being made to bring out the products of these groups in solid form. Several are already marketed in the powder form, as well as in paste. It would be well if those not yet appearing in powder form could also be thus marketed, since pastes easily dry out, and the weighting out, or the determination of the amount of dyestuff, is made difficult. The advantages of the powder form are easily apparent. So far one part of powder is equivalent to two parts of paste. The products are easily soluble in water.

The powders are generally pasted up with water at the ordinary temperature, the paste further diluted, and poured through a fine sieve or strainer into the dye bath, which is made up to 100 liters with 200 to 300 g. of Marseilles soap or Monopol soap. Soft water is the best to use. In general, black is dyed at 75° to 80° C., but with the lighter colors dyeing is begun at a lower temperature. For Cellitone Fast Yellow G powder, RR powder, and Cellitone Red B powder, the dystuff is stirred, for making up, into water of about 40° C. Further details are as given above.

3. *The Cellite Azoles.*—In these dyestuffs, which are diazotized and developed, we possess principally lively red tones, Bordeaux, a few reddish yellows, a copper, a dark-blue, and a black. We are, therefore, somewhat limited in possibilities for combinations more especially because, with the different dystuffs, we are compelled to use certain developers for certain tones. As an example, we may take Cellite Azol R. For this dyestuff, using phenol as developer, we get a reddish-yellow; resorcin gives a yellowish-red, beta-naphthol a pure bluish-red, Developer B or ON give Bordeaux.

The getting of the Cellite Azoles into solution varies in method according to the brand. Cellite Azol SR and B are boiled up for a short time with boiling-hot water, as soft as possible, to which has been added hydrochloric acid or Tetraline, soap, and soda; while Cellite Azol ORB or ST are simply dissolved in hot water. In the dyeing, too, there are differences; Cellite Azol ST, R, and BB are dyed without any admixture, while with Cellite Azol ORB Glauber salt and acetic acid are needed. There are other differences, too, which have to be learned from the detailed color-cards and directions for use.

In the diazotization we find fewer differences. The baths are used cold, and in general we employ, on the weight of the goods, 4% of sodium nitrite and 10% of hydrochloric acid of 20° B.; for Cellite Azol R, 6% nitrite and 15% acid. In general, for long working, at least 2 g. of nitrite and 5 c.c. of acid are added per liter. After diazotization, the goods are rinsed at once, and developed. The developers used are dissolved in various ways, and care has to be taken in using them. Development itself is carried out usually as in cotton dyeing, in a cold or luke-warm bath; only Developer ON behaves otherwise, and must be used at 60° to 70° C. If this developer is gotten into solution with soda or caustic, enough acetic acid must be added to the bath to make it weakly acid.

Acetate Rayon in Mixed Weaves

In considering this kind of fabric, it is obvious that here, too, the Cellite Azoles can be used, especially when it is a question of getting dark tones of good fastness to rubbing. Naturally, two-color or polychrome effects can be attained more simply by using the Cellite Fast dyestuffs or the Cellitone or Cellitone Fast dyestuffs, which will be mentioned only incidentally. The Cellite Azoles probably possess the greatest value for dyeings fast to washing, or for those which are to be overdyed.

4. *Wool Dyed, Acetate Silk White.*—Since acetate silk loses luster and strength by treatment at the boil, 85° C. is taken as the highest limit of temperature. Dyeing is carried out with addition of 10% of Glauber salt cryst., and 3% to 5% of formic acid, and the goods are well rinsed after dyeing. Some of the dyes which can give

many mode tones, etc., are Flavazine S, Supramine Yellow R, Fast Light Orange G, Alizarine Rubinol R, Radio Red VB, Alizarine Direct Violet EFF, Anthracyanine 3GL, Cyananthrol BGA, Fast Acid Marine Blue HBB, Anthracyanine Brown RL, Radio Brown B, Anthracyanine Green 3GL, Guinea Fast Green B, Naphthylamine Black EFF.

5. *Wool Undyed, Acetate Rayon Dyed.*—Dyeing is carried out with the Cellitone or Cellitone Fast dyestuffs, with addition of Dekol or soap. The wool is more or less tinged (angeschmutzt) according to the dyestuff; one cannot speak of the wool remaining actually truly undyed.

6. *Wool and Acetate Silk Dyed, Two-Color Effect.*—The dyestuffs given under 4 and 5 are used, dyed with addition of Glauber salt and acetic or formic acid (see 4). Or, the dyestuff for the acetate silk can be applied first at 50° to 60° C., then the wool dyestuff added to the bath, and the dyeing completed at 85°. (Cellitone Red R, Cellitone Fast Blue BB, Cellitone Fast Marine Blue BB and G in paste, Cellitone Fast Black B and G in paste, are less suitable for two-color effects, since they dye the animal fiber too strongly.) The tinging of the wool, mentioned above under 5, plays no unfavorable part in the production of two-color effects, if it is taken into consideration in deciding upon the tones desired.

7. *Cotton or Viscose (or Cupro-Silk) Dyed, Acetate Silk Undyed.*—Dyeing is carried out at 60° to 70° with substantive dyestuffs, with addition of 10% to 30% of Glauber salt cryst., or with soap and Glauber salt. Suitable dyestuffs are Sirius Yellow R extra, Sirius Orange G, Sirius Brown R, Sirius Pink BB, Sirius Rubine B, Sirius Violet BB, Diamine Pure Blue FF, Sirius Blue BRR, Sirius Green BB, Sirius Gray G, Diamine Black RMW.

8. *Cotton, Etc., Undyed, Acetate Silk Dyed.*—The Cellite Fast, or the Cellitone or Cellitone Fast, dyestuffs, are used as under 1 and 2. After dyeing, the goods are washed in a weak soap bath. (The Cellitone or Cellitone Fast dyestuffs leave the cotton most white.)

9. *Cotton, Etc., and Acetate Rayon Dyed, Two-Color Effects.*—The dyestuffs mentioned under 7 and 8 are used. The Cellitone Fast Marine Blues, mentioned under 6, as not suitable there, may here be employed. With mixtures with the Cellite Fast colors, dyeing is carried out under addition of 20% to 40% of calcined Glauber salt; with the Cellitone and Cellitone Fast colors (bath ratio 1-25 to 1-30), with 7.5% to 30%, and also 5% to 6% of a neutral soap, at 70° to 75°. The one-bath process is here being considered. According to the two-bath procedure, we get still purer dyeings by first dyeing the acetate rayon, washing well in warm water, and after dyeing the cotton is in a fresh bath with addition of Glauber salt at 40° to 50°.

10. *Half-wool Dyed, Acetate Rayon Undyed.*—Dyeing is carried out at 85° at the highest, with addition of Glauber salt. At a bath ratio of 1-20 to 1-25, 10% to 40% of Glauber salt cryst. on the weight of the goods is needed, according to the depth of shade wished. Dyestuffs used are the substantive ones, and likewise such acid dyestuffs as go on in neutral bath, which will leave the acetate rayon clean white. Some suitable dyestuffs are Chrysophenine G, Benzo Fast Orange WS, Sirius Pink BB, Sirius Scarlet B, Sirius Brown R, Chloramine Violet FFB, Benzo Chrome Black Blue B, Union Green KB, Cotton Black A4G, Diamine Black BH. Some suitable acid dyestuffs are Fulling Yellow O, Acid Anthracene Red G, Formyl Violet S4B, Patent Blue A, Tolyl Blue NB, Alizarine Cyanine Green G extra.

To dye the acetate rayon, and to leave the cotton, etc., uncolored, is a problem not often occurring, but often the following combination is desired:

11. *The Different Fibers in Either Two or Three Color Effect.*—For two-color effects the union element is dyed with addition of 10% to 20% of Glauber salt cryst. To protect the wool about 2% to 4% of Protectol Agfa II is added, which may also be done under 10 (above). The goods are entered at a low temperature, and dyed at 80° to 85°. Beside the dyestuffs mentioned under 10, the following are also suitable: Diamine Fast Yellow A, Diamine Orange D, Diamine Heliotrope B, Sirius Blue 6G, Sirius Gray R, Oxy-Diaminogen OT. As acid dyestuffs which go on in neutral bath, there may be recommended the following: Sulphone Yellow 5G, Wool Fast Violet B, Alizarine Pure Blue B, Alkali Fast Green 10G. After dyeing the goods are rinsed, and the acetate silk after-dyed with Cellitone or Cellitone Fast dyestuffs. Recently the wool and acetate silk are being dyed first in a single bath for two-color effects, and then the cotton after-dyed, with addition of Katanol WL.

Handsome two-color effects can be gotten by dyeing the half-wool element with substantive dyestuffs and neutral bath wool dyestuffs to the same tone, and then dyeing the acetate rayon element as desired with Cellitone or Cellitone Fast dyestuffs. Dyeing is carried out in a neutral Glauber salt bath, raising the temperature (on account of the wool) to 80° to 85°, dyeing for ½ to ¾ of an hour, and then for the same time in the cooling bath, in order to cover the cotton well.

12. *Three-Color Effect on Union Goods and Acetate Rayon.*—These goods are dyed either in one bath, or better in two baths. If dyed in one bath this is done with substantive dyestuffs and neutral-bath wool dyestuffs, with addition of Glauber salt (dyestuffs which leave the acetate rayon untouched as far as possible), or, further, with the Cellitone or Cellitone Fast dyestuffs. With the two-bath method, the wool and acetate silk are first dyed in a weakly acid bath, with Glauber salt, at 80° to 85° C., then the goods washed, and the cotton after-dyed in a fresh bath, at about 50°, with addition of 20% of crystallized Glauber salt and 3% of Katanol WL, to the desired tone. It is believed that the two-bath method makes possible a better dyeing or shading of the acetate rayon. Suitable dyestuffs can be selected from the lists

already given in preceding paragraphs. A few other substantive dyestuffs, which are suitable for the Katanol procedure, may be mentioned: Direct Yellow R extra, Sirius Orange G, Benzo Fast Scarlet 4BS, Brilliant Benzo Violet BBH, Diamine Pure Blue FF, Benzo Blue BB, 3B Diamine Black BH, and Direct Black VT.

13. *Natural Silk Dyed, Acetate Rayon Undyed.*—Dyeing is carried out as under 4, that is, in acid bath. Some suitable acid dyestuffs are: Fulling Yellow O, Sulphone Orange G, Amidonaphthol Red G, Radio Brown B, Formyl Violet S4B, Brilliant Fulling Blue B, Sulphone Cyanine GR extra, Alizarine Cyanine Green G extra, Radio Black ST.

14. *Natural Silk Undyed, Acetate Rayon Dyed.*—The acetate rayon is dyed as described under 2. The temperature of 85° C. should not be exceeded. Since the natural silk is mostly colored somewhat, rinsing warm or slight washing in a soap bath is required.

15. *Natural Silk and Acetate Rayon, Two-Color Effect.*—The two-color effect can be gotten by the one-bath procedure, but the purest shades by the two-bath method. In the one-bath procedure, dyeing is carried out with the Cellitone or Cellitone Fast dyestuffs, and the acid dyestuffs, dyed in an acid bath as under 13, or else in a Glauber salt-soap bath with suitable substantive neutral-bath acid dyestuffs. If dyeing is carried out at 50° to 75°, with addition of 20% to 30% of Glauber salt and 3% to 5% of Marseilles soap, dyestuffs suitable for use in combination with the Cellitone or Cellitone Fast dyestuffs are, among others: Chrysophenine G, Diamine Fast Orange EG, Brilliant Geranine B, Diamine Brilliant Rubine S, Sirius Brown R, Brilliant Benzo Fast Violet RRL, Diamine Brilliant Blue G, Brilliant Benzo Green BMAs acid dyestuffs used in neutral bath, the dyestuffs mentioned under 13 (except Amidonaphthol Red G and Radio Black ST) may be used; also, for red, Acid Anthracene Red 3BL; for blue, Wool Fast Blue GL, for green, Brilliant Acid Green 6B; for black, Sulphone Cyanine Black BB.

If the two-color effect is to be gotten by the two-bath method, the acetate rayon is dyed as given under 2, the material then washed, and the natural silk after-dyed, at a temperature not too high, with the acid dyestuffs given under 4 and 13.

16. *Natural Silk, Viscose, and Acetate Rayon, Two or Three Color Effects.*—By a two-color effect we usually understand the dyeing of the natural and viscose silks in the same tone, and that of the acetate rayon in another tone. Viscose and acetate silk elements are dyed together, with addition of 5% to 20% of Glauber salt cryst. per liter of bath, at 30° to 50° C., with Cellitone or Cellitone Fast dyestuffs, and suitable substantive dyestuffs, and the natural silk element dyed in a fresh and acid bath, at 50° to 60° with acid dyestuffs. Here, too, the natural and acetate silks can first be dyed, as mentioned under 11, and the viscose after-dyed.

For three-color effects, the order of dyeing is: first the acetate rayon, then the viscose, and finally the natural silk. A dyer with a great deal of experience can also produce a three-color effect in one bath, dyeing with addition of Glauber salt and soap. For example, in making up the bath, Alizarine Cyanine Green 3G and Sulphone Yellow R are used for the natural silk (olive), Cellitone Fast Red Violet R for the acetate silk (heliotrope), and Sirius Orange G for the viscose (orange).

17. *Wool, Natural Silk, Cotton or Viscose or Cupro-Silk, and Acetate Rayon Four-Color Effects.*—Production of a four-color effect is theoretically possible, but practically it is almost a piece of dye jugglery, which is better left undone, and the desired effect gotten by previous dyeing of at least one element in the yarn stage (e. g., black). In piece goods the method of working might perhaps be about as follows: the wool and acetate rayon elements first dyed in a two-color effect in one bath, in carrying out which dyeing such acid dyestuffs would be chosen as would leave the natural silk as clean as possible in the acetic acid bath. Then, dyeing in a bath containing Glauber salt (with addition of Katanol WL, to avoid the dyestuff taking upon the wool and natural silk) on the cotton or viscose, with substantive dyestuffs; finally, in a fresh bath, at a low temperature, with addition of acetate acid, dyeing the natural silk with the previously-mentioned dyestuffs.

After-Treatment of Azoic Colors

Effect on Fastness Properties

THE question of the "blinding" of artificial silks is a subject which has received an important amount of attention both in this country and on the Continent, since F. M. Rowe first drew attention to the phenomenon a year or two ago. Dr. Rowe showed that when artificial silks were dyed with the insoluble azo colors the masking of the luster of the fiber was due to the coagulation or the crystallization of the coloring matter into larger aggregates within the fiber, brought about by moist heat. Before proceeding to indicate the extent and details of some new work carried out by the above-mentioned author along with Dr. Percy Bean, Jr., reference is made to the work which has already been published or is already known from works' practice.

It has long been known that aftertreatments do influence the shade and fastness properties of materials dyed with pigment colors, and as a result it has been usual in work to soap materials dyed with the earlier insoluble combinations such as Para Red, in order to get better fastness by rubbing and to obtain a brighter shade by removing loose surface coloring matter.

It has also been suggested to give an aftertreatment with calcium chloride solution to lessen the bleeding of these colors, while boiling with solutions of these salts—e.g., copper sulphate—with or without increased pressure, has been recommended for increasing the fastness to light.

Such treatments can scarcely affect these coloring mat-

ters chemically, and any material influence which they exert must be due to an alteration in the physical condition of the coloring matter. As the members of the Naphthol series became more widely used, it was found that hot soaping primarily intended for improving the fastness to rubbing also altered the shade and increased the fastness to light of many of their azo combinations.

An example of the fact that the shade and properties of material dyed with pigments are largely dependent upon the physical condition of the coloring matter in the fiber has long been provided by Indigo. Thus the red tone of some Indigo dyeings, originally attributed to the presence of Indirubin in Natural Indigo, is due to the presence of crystals of Indigotin. This crystallization of indigotin, with consequent reddening and bronzing of the dyeing, is readily brought about by steaming the dyed material, but at the expense of the fastness to rubbing. Many variations in the shade and fastness of other vat dyes encountered in practice are also due to variations in the degree of aggregation of the coloring matter in the fiber.

Other examples of changes of shade, which indicate changes in the physical condition of coloring matter in the fiber, are:

1. Those which occur when material dyed with some insoluble azo compounds is dried over drums, e.g., Para Red assumes a yellower tone when hot, becoming slowly blue on cooling, and,

2. The color changes of dyes, e. g., Diamine Blue 3R, which are not fast to ironing.

Contemporary Work and Results

Loechner had examined the comparative effects of water, soap solution and caustic alkaline Turkey Red oil at various temperatures up to 100° C. and for periods up to half-an-hour on the fastness to light of three insoluble azo combinations dyed on bleached calico, and established the fact that the fastness to light is increased by suitable aftertreatment. Excess Naphthol or excess diazo-compound does not influence the fastness to light and the maximum increase in fastness to light is purely a function of the more or less intensive hot aftertreatment of the material. With certain combinations of Naphthols great importance is attached to a bailing aftertreatment, while with other combinations there is much less effect in increasing the fastness to light. The combination Fast Red RL-Naphthol AS-RL as produced on the fiber at first usually appears to contain a yellowish impurity, which affects the fastness to light adversely unless removed by soaping; the resulting shade is brighter and faster to light.

Even boiling in water for five minutes considerably increases the fastness to light of the combination Fast Red GL-Naphthol AS, whereas boiling in soap solution for half an hour is necessary to increase considerably the fastness to light of the combination Fast Red KB-Naphthol AS-SW.

Loechner draws the erroneous conclusion (in the opinion of Rowe and Bean in the current issue of the Journal of Society of Dyers and Colorists) that only hot, faintly alkaline aftertreatments materially influence the fastness to light, whereas the correct conclusion is that the use of alkali and soap brings about the maximum increase in fastness to light more rapidly than is the case in their absence at ordinary pressure.

Aggregation of Dye Particles

Kayser was the first to connect the increase in the fastness to light of aftertreated dyeing on ordinary and mercerized cotton with a change in the degree of aggregation and location of the coloring matter in the fiber. He stated that microscopic examination of material dyed with azo combinations of the Naphthol AS series shows that in untreated dyeings the coloring matter is always located in the cell wall, whereas in dyeings aftertreated in hot water or boiling soap solution the coloring matter is located in the lumen.

Certain combinations indicated lowest fastness when only rinsed, while those soaped boiled had the highest fastness.

The aggregation and wandering of the coloring matter articles is closely connected with the swelling of the fiber and reaches a maximum with aftertreatments, which promote swelling—e. g., boiling alkali or soap—whereas common salt solution at 109° C. prevents swelling and no migration of the coloring matter occurs.

Effect of Fiber Swelling

Ruperti's work showed that the aggregation of coloring matter by the action of moist heat occurred in greater or less degree with all types of dyeings, which he examined on cellulosic fibers, but the conditions necessary to bring about distinct aggregation varied considerably with individual cases. Differences in fastness to light due to changes in the degree of aggregation of the coloring matter consequent upon aftertreatment were found in most, but not all cases. This author holds the dyeing of cellulosic fibers to be a precipitation of coloring matter partly within the fiber in a very finely divided form, and partly on the fiber where it is retained by absorptive power. When a coloring matter is aggregated in the fiber by hot water or steam, swelling of the fiber is an important and probably decisive factor in allowing the pigment particles sufficient freedom of movement to form coarser particles in the cell wall.

As regards the change in shade of a dyeing, this only takes place when the degree of dispersion of the coloring matter in the cell wall is affected, and this needs moist heat. Although moist heat influences the shade and fastness properties the coloring matter is not altered chemically as is shown by examining the absorption spectrum before and after treatment.

Other investigators have also shown that changes in shade produced by moist heat are due to increase in the size of the pigment particles, while changes in shade in the reverse order produced by dry heat at 250° to 300° C. are due to decrease in the size of the particles. With the Azo compounds of the Naphthol AS series moisture is essential for aggregation to occur, although only a mere trace is quite enough, and moist heat increases the fastness to light, while dry heat decreases the fastness to light.

Recent Work

In the present work of F. M. Rowe and Percy Bean, Jr., considerable steps forward appear to have been made towards a better and one almost feels a final understanding of what is happening in the dyeing of the Naphthol AS series of colors on the vegetable fibers, like cotton and artificial silks, and the condition of the dyed fiber after it has received it various processes subsequent to dyeing. There appears to be solid ground for the reasons advanced concerning the why and wherefore of the fastness to various influences. These authors found that hot aftertreatments influence the state of aggregation and location of azoic colors in the fibers in varying degree according to the nature of the dyed fiber, the constitution of the azo compound, the depth of shade and the severity of the treatment employed.

Interesting details of what happens within the fiber, the best aftertreatments to give Azoic-dyed materials, and the effect on fastness properties will be dealt with next week—*Textile Argus.*

PLANS UNDER WAY FOR 1930 SOUTHERN TEXTILE EXPOSITION

The work of allotting space to exhibitors for the Ninth Southern Textile Exposition, to be held in October, 1930, has begun. Although the exposition is a year off, nearly all the floor space has been applied for. One hundred and fifty exhibitors are now enrolled.

W. G. Sirrine, president, stated that it would be the best organized and equipped exhibition ever held in Textile Hall. Those who fail to file applications at once will find it hard to get reservations, as there remains only a few sections on the four floors of the building. Every part of Textile Hall will be used, including the space adjoining the boiler-room.

As usual the annex and main building will be connected by temporary passageways. Balcony space will be more advantageous than in previous shows due to recent alterations. There is now a continuous aisle around the balcony affording room for several booths over the stage. Repainting and repairs are already under way. Considerable concrete work is being done to improve the drainage in the rear of the buildings.

All the leasing manufacturers of looms will make displays. Spinning machinery will not be shown, but everything else about a cotton mill will be seen.

Laundry Processes Improved by Studies of Effects of Cleaning Agents and Methods

By Henry D. Hubbard

Assistant to the Director, Bureau of Standards

THE Bureau of Standards is technically concerned with the "refreshment" of clothing, the new word for laundry processes which restore garments to wholesome cleanliness important for hygiene, good appearance, and garment conservation. "Refreshment" includes removal of stain spots, restoration of the nap and "feel," bleaching, ironing, to restore the fabric to initial freshness. The Bureau's well-equipped laundry research laboratory undertakes the experimental search for cleaning agents for any stain on any fabric. A great machine industry has developed from the old-home industry—the family wash.

Soap is an index of civilization. We extol its virtues in print by ad and text, by radio, in school; everywhere the cult of cleanliness is becoming almost a sacrament. A new stage appears when science enters the field. Each ingredient and process detail must qualitatively and quantitatively justify itself.

The Bureau of Standards Circular on "Soap" (available by purchase only) outlines manufacturing processes, and, from the countless varieties, found seven groups adequate to classify soaps by their service function. A discussion of the composition of each group is given and the circular is a document full of information. A revision has just been completed and will soon be sent to press. A discussion of composition, ingredients and specifications is given which became the basis for the Federal specifications for soap for Government use.

Other cleansing agents are described in brief in the well-known Circular 70, Materials for the Household (available by purchase only), issued by the Bureau for households and teachers and students in domestic science.

By act of Congress research facilities of the Government may be made available to qualified workers. Under this act the Bureau of Standards research associate system was established, and its marked success is a notable example of the Bureau's co-operation with industry on its basic problems.

The Bureau's unique research equipment in some seventy specialized sections is made available for industries which have no research laboratories and to supplement those which have already some provision for research. The researches are usually planned jointly by the Bureau and the technical committee of the entire industry.

When the National Association of Dyers and Cleaners expressed the desire for such co-operative research in a systematic program, the Director of the Bureau appointed a technical expert of the industry as research associate, maintained by the industry but working under the supervision of the Bureau experts and administration.

There resulted from the work of this associate a reference book of the cleaning art, compiled with the full co-operation of Bureau experts; a series of short-term schools for managers and foremen in various parts of the country on the latest advances in the cleaning art; and eventually a special research laboratory built for the industry, near Washington, and designed to work in close harmony with the Bureau of Standards.

Dry cleaning—by the use of organic solvents—avoids shrinkage caused by water washing and involves less rubbing. The disadvantages, until recently, were the explosion hazard and the grease remaining in the garment. The machines are now gas-tight and electrically grounded to avoid explosions from static sparks. So gas-tight are modern plants that the air is as sweet as in any other type of building.

The Bureau of Standards aided notably in the development of the Stoddard solvent by experimental determination of the limiting amounts of highly volatile gasoline, thus minimizing hazard, and by determining the limits of the nonvolatile oily constituents, thus minimizing the grease remaining in the garment after dry cleaning. Methods of inspection and test were worked out to insure efficiency and safety and the new Stoddard solvent is now being produced by various manufacturers. The name is from a leading industrial expert who was active in developing its use.

The Bureau of Standards used its good offices through the procedure for adopting commercial standards, with success, so that the cleaning industry has adopted the specifications as a national commercial standard for the industry.

An excellent type of research was the reclamation of dry-cleaning gasoline. At the time 45,000,000 gallons were being used annually—clearly an item worth saving. After a study of methods proposed and in use, a modified method was developed in detail to save the gasoline otherwise lost by evaporation or wasted by lack of means to purify it. The research associate with the co-operation of other members of the staff brought the research to such success that the reported annual saving to the industry touches the seventh figure.

The Bureau found that most dyes are not affected by dry-cleaning solvents. To minimize the effect on susceptible dyes, it was found that the cleaning agent should be water free and neutral—that is, not alkaline. Substitutes for susceptible dyes are being sought. A large number of different dyes were tried on a variety of fabric, cotton, silk, rayon, and wool and the effect was found nil. This gives greater assurance to the public that dry cleaning will not injure delicately dyed garments.

It was found that silks deteriorate in light more rapidly when weighted with tin (absorbed by the silk fibers as a salt of tin). At times they completely disintegrate. Dry cleaning, after investigation, was acquitted as not guilty of the damage. Hence the cleaner is justified in not assuming the risk, since he does not and cannot easily learn the previous history of the garment. It was disclosed that garments which appeared to be in good condition when they arrived at the cleaners may, on coming out, collapse into a powder when handled. The damage to silk caused by light was found to be greater when the silk was wet with perspiration. A research is in progress on the effect of diffused light indoors on unweighted silks and on silks variously weighted, since it is already known that excessively weighted silks lose strength faster in sunlight.

Many garments having fur and leather trim are dry cleaned. Such trim is filled with oil to make it pliable. The solvent used in cleaning dissolves out the oily finish material leaving the fur or leather brittle.

The Bureau of Standards workers developed a means to avoid such damage. By adding paraffin, the "fat-liquor" so essential to pliability is kept intact, and the appearance and in some cases the luster is improved. The addition of naphtha prevents attack by organisms and furnishes a cheap, colorless material which does not discolor nor become rancid. The research gave the answer to the gain of the industry and the public.

Performance tests of a liquid laundry soap in washing textile materials were published, giving the peculiar merits of the soap: Less shrinkage in washing in some classes of fabric, less injury from repeated washings, better "feel," slighter change of color, greater resilience in washed wool tops, and better scouring and enhanced whiteness on fine-grade wool fleece.

A typical research is the discovery of the cause of the so-called "winter damage" to textile goods laundered in the winter time. It was found that the damaged goods always contained sulphuric acid and had been washed and dried out of doors. Atmospheric sulphur dioxide from winter fires became suspected as the offender.

The decisive test proved this to be the active cause. This test was to wash a number of towels repeatedly outdoors and iron them by hand. These lost breaking strength faster than when washed and ironed in the laundry. This loss in breaking strength was then duplicated by exposing damp towels to air containing one-millionth sulphur dioxide. The chain complete, it was found that the sulphuric acid formed in the damp fabric was notably active at the temperature of the hot iron used in ironing the towels. The remedy devised was to produce calcium bicarbonate in the final rinse—an alkali which is a natural neutralizing agent for acid. The remedial process has been used in New England laundries with great success in minimizing the "winter damage."

On the program of the Bureau for cleaning research is the elimination of prespotting. At present each spot must be removed by an appropriate reagent. It is hoped to introduce into the cleaning bath such suitable reagents as will remove the necessity of individual handling of garments and the prespotting of the individual stains. This is a look ahead full of promise for successful research.—*The United States Daily.*

COMMISSIONER DAUGHERTY REPORTS SATISFACTORY CONDITIONS IN GERMAN CHEMICAL TRADE

Germany's favorable foreign trade balance in chemical products in the first six months of 1929 was $14,000,000 in excess of the corresponding 1928 figure. Imports of commodities classified as chemicals in German statistics amounted to 950,693 metric tons in the six months ending June 30, 1929, while exports totaled 2,691,989 tons, making an excess export figure of 1,741,296 tons against a corresponding 1,531,790 tons in the first six months of 1928. The values for the first six months of this year were 162,498,000 marks for chemical imports and 683,196,000 marks for exports, against 160,752,000 and 622,849,000 marks respectively, a year previous. A favorable trade balance of 520,698,000 marks, therefore, was established in 1929, as compared with 462,087,000 marks in the corresponding 1928 period.

The 10% gain in exports represented increased sales of nearly all commodities, and an examination of import statistics of individual classes shows few significant changes. Such important items as benzol, rosin, and phosphate rock are not included in the German chemical classifications.

With the exception of coal and potash, in which Germany is more than self-sufficient, most of the minerals and other raw materials required for the chemical industry must be imported. Sulphuric acid is made from American sulphur and from German, Spanish and Scandinavian pyrites. Copper, zinc, lead, tin, nickel and aluminum metals and salts are produced largely from imported ores. The United States is prominent as a source of borax and phosphate rock as well as of petroleum. Over 65% of naval stores imports are of American origin.

Coal-tar dyes, the second most important group in German chemical exports, were somewhat larger in volume, but smaller in value, than during the first six months of 1928, as shown by the following table:

GERMAN COAL-TAR DYE EXPORTS

ITEM	FIRST SIX MONTHS OF 1928 Quantity Metric Tons	1928 Value Thousand Marks	FIRST SIX MONTHS OF 1929 Quantity Metric Tons	1929 Value Thousand Marks	To the U.S., First 6 Months of 1929 Metric Tons
Anilin dyes ..	16,010	85,308	14,998	75,018	607
Alizarin dyes .	1,152	14,905	1,329	16,756	468
Alizarin red ..	1,095	2,348	1,153	2,253	.....
Indigo	6,197	16,470	7,741	17,278	130
Indigo carmine.	152	435	.....	.....	.....
Total ...	24,606	119,466	25,221	111,305	1,205

Of the 112 chemical firms reported on in 1928, only 20.5% passed dividends as compared with 29% in 1927, and 56% in 1926. The average 1928 interest yield on capital was 9.9%, in contrast to 6.6% in 1925. Excluding the I. G. with its 12% dividend, the average is reduced to 7.1% for 1928, almost double the corresponding 1926 rate (3.7%).

Since May, 1928, employment in the chemical industry has been maintained at a higher level than for German industrial activities as a whole, fluctuating between 89 and 95% of the trade-union membership and ranking 3.5% above the average for all industries in May of this year.

ESTIMATION OF RAYON IN MIXTURE FABRICS

Dealing with methods used for estimating the amount of rayon in mixture fabrics, a writer in the current issue of the *Artificial Silk* (Rayon) *World* says:

"The usual methods for the analysis of union fabrics are not applicable to materials containing the regenerated cellulose artificial silks, and on account of the ever-increasing use of the artificial silks in almost all kinds of fabrics new methods of analysis are necessary. The necessity for a suitable analytical method is evident from the duty imposed on rayon in most countries, and where the textile methods of dissection are not applicable the percentage of artificial silk has been very difficult to determine. The rayons were first employed alone, or as warp or weft yarn, or twisted with wool, silk or cotton. The ingenuity of the designer or manufacturer has produced fabrics from materials of various kinds that are too difficult to dissect to arrive at their relative fiber composition.

Cotton and wool mixtures may be readily separated quantitatively by boiling the weighed material with a 2% solution of caustic soda, the wool being dissolved and the cotton scarcely affected. This process cannot be applied to a mixture of wool with regenerated cellulose rayons, however, because the alkali causes a loss in weight of the rayon on account of solvent action. P. Krass and E. Biltz, *Textile Forschung*, 1920, II, 24, recommend the use of ammoniacal copper solutions to dissolve the rayon for such analyses. According to their method about 0.2 to 0.5 gram of the sample is weighed and treated in a porcelain dish with 10 c.c. of ammoniacal copper solution for about half an hour with occasional stirring. The solution is decanted, the residue being treated for a further half hour with fresh ammoniacal copper, and the solution again decanted. The residue, after being washed with strong ammonia, is then well washed with water, with 10% hydrochloric acid, and with cold and warm water, being finally pressed between filter paper and dried at 110° C. Wool treated by this method loses 0.42% in weight. These authors state that the cuprammonium silk loses 6% and viscose silk 7% by weight when treated with caustic alkali of the concentration that is employed to dissolve wool from a mixture of wool and cotton. Lloyd and Priestley found very variable figures for the loss in weight when different regenerated cellulose rayons were boiled with either 1% or 2% caustic alkali solutions.

The ammoniacal copper method of analysis is not applicable to the analysis of materials containing cotton along with rayons and animal fibers.

After removal of the rayons in mixtures of artificial silks, cotton and wool, the cotton may be removed by Schweitzer's reagent and the wool weighed. The cotton may also be removed by the prolonged action of sulphuric acid of 69° Tw. The material from which the rayon has been removed or a weighed portion of fresh material is heated to 50° C. with the acid and left, preferably in a temperature of about 25°-30° C., for 24 hours. Vegetable fibers become completely hydrolyzed into water-soluble products. The acid is decanted off and the wool remaining is well washed with water, dried, conditioned as with original material and weighed.

The following are analyses of various materials. The method was tested by weighing separate fibers and then analyzing them after mixing.

Fibers	Weight Taken	Weight Found	% Gain or Loss
Cotton	3.2	3.36	+5.0
Botany	2.4	2.26	—5.8
Viscose	5.6	5.58	....
Cotton	3.5	3.46	—1.1
Botany	2.5	2.46	—1.6
Viscose	5.2	5.28	+1.5

Material	Wool	Viscose
Botany Viscose Serge	62.2%	30.8%
Botany Viscose Serge	51.3%	48.7%
Botany Viscose Top	70.1%	29.9%
Botany Viscose Top	61.2%	38.8%

	Chemical Analysis	
	Wool	Viscose
Twisted Yarns of Viscose Worsted	69.7	30.3
Ditto (softly twisted)	63.6	36.4
Viscose and Wool Fabric	74.5	25.6

	By Dissection	
	Wool	Viscose
Twisted Yarns of Viscose Worsted	68.7	31.3
Ditto (softly twisted)	62.0	38.0
Viscose and Wool Fabric	74.4	25.6

	Wool	Cotton	Viscose
Cotton, Wool and Viscose fabric	43.3	30.2	26.5
Ditto	48.0	24.9	27.1

	Wool	Cotton	Viscose
Cotton, Wool and Viscose fabric	44.3	31.0	24.7
Ditto	46.9	26.3	26.8

—*Artificial Silk World.*

It is reported from Northboro, Mass., that negotiations are under way for the leasing of a plant in that place for the manufacture of rayon goods.

EXTRACTORIALS

Lack of bad habits does not necessarily predicate the possession of good habits, and if it is honestly desired to get something more out of life than a mere existence, there must be followed a code of positives—a form of goodness which does things. Too many of us are prone to play safe by doing nothing, taking pride in our lack of bad habits, smugly assuming an unwarranted sense of superiority because of such lack, and thanking God that we are not as other men.—*The Kalends.*

In the present era of industrial development the struggles of unfavorably situated enterprises merely prolong the agonies of their economic destruction. Consequently, when consolidations are acknowledged to be economically sound, and when the prosperity of a large group of the industry is dependent upon the merger of several interests, mill owners and executives should have no hesitancy in sacrificing personal glory and individual authority for the ultimate salvation of invested capital.—*Textile World.*

Army advances depend upon material as well as personnel. A break in the service of supplies—too great a gap between men and munitions—means slow progress if not ultimate defeat.—*Chemical and Metallurgical Engineering.*

ENGLISH FORM COLOR COUNCIL

There was established on October 9, 1929, a British Color Council at an inaugural meeting of between one and two hundred representatives of almost half a hundred trade associations which covered diversified branches of the industry.

There was almost a constant reference to the position which the United States was holding in respect to the issuance of color cards. It was generally expressed that Europe was realizing a greater and greater tendency towards following American colors, especially in the manufacture of millinery.

Speakers stressed the great need of Britain, namely the standardization of color names and the abolishment of misleading descriptions which do not suggest individual shades.

A. A. Thomson, American counsul at Bradford, assured the British manufacturers of the whole-hearted support of United States. The reading of a cable of good-will from Margaret Hayden Rorke, managing director of the Textile Color Association of the United States, Inc., was greeted with cheers.

Deprecation of British manufacturers' effort in developing new styles in colorings for women's dress goods by Edward Symonds, of Reville dressmakers, brought spirited replies from manufacturers who called for cessation of unwarranted tirades of pessimism regarding Britain's initiative courage in this direction.

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PERSPIRATION—A TESTING PROBLEM

MOST of the tests for dye fastness proposed by technical associations and trade groups are not yet definitely established. Some of the so-called standard tests are still in the experimental stage. The notion that fastness has been completely standardized is a harmful notion because it may weaken the general interest in research on fastness tests. It is a notion that may lead many chemists and mill men to believe that no problems remain. It may deter them from co-operating in the good work of financing fastness research. The unsolved problems are often the most important.

Fastness to light, it is true, has been studied so thoroughly both by American and British societies that standard tests should soon be laid down—at least in the United States where the Research Committee of the A. A. T. C. C. has worked so conscientiously to unravel the secrets of light fading. Washing tests have been standardized for almost every condition, and a laboratory machine for testing invented and adopted. But there are other color-destroying agents which will continue to baffle dye and textile chemists for some time to come. Perspiration and its effect on color and finish is one of the unsolved problems of fastness research.

Perspiration is an agency difficult to study because it cannot be easily controlled. Light—even sunlight—is so nearly mechanical in action over a given period of seasons that laws of fading can be established, involving even such variable factors as humidity. Washing is obviously a combination, basically, of mechanics and soap chemistry, and testing methods for use with the Launder-Ometer or other laboratory washing machine now present very few problems. But when you come to perspiration it must be recognized as a peculiarly *personal* color-destroying agency. It may vary almost to the extent that human character varies. There can therefore hardly be a standard perspiration. A dozen standard types may be necessary. The factor of time enters to complicate tests, for the chemical constitution of perspiration will change on standing.

"The ammonia nitrogen content was found to increase on standing," states a recent comment in Textile Maintenance Notes.* "This probably explains why some investigators found perspiration alkaline. Their examination was probably made some time after the sweat was given off with the result that it analyzed alkaline. The rapid rise in ammonia content of perspiration was found to be due to the presence of an activator, bacteria normally present on the skin. This leads to the conclusion that in any problem regarding the fastness of dyes to perspiration, regard must be paid to the changes of the constituents of sweat due to the bacterial infection of any garments worn near the skin. Much of the importance of these experiments lies in the fact that they may permit the development of a synthetic perspiration by means of which dyes and fabrics may be tested before being marketed."

Despite the tests for fastness to perspiration that may now be in use, it is evident from this opinion that the subject calls for further study of a painstaking nature. Some of us may consider perspiration as a minor agency in fastness research, but it may yet be disclosed as a problem far more difficult than the standardization of light fading.

WHY THESE MERGERS?

WE are hearing a deal of comment to-day about the increasing number of mergers in industry. As in the old trust-busting era the subject has become front-page news. It has been loosely styled by some as a craze, and the fact remains that there is an element of contagion in the movement which almost makes it appear like an epidemic. This view of mergers, however, might seem to brand them as an abnormal malady and therefore dangerous to the public or to industry. Queerly enough, the question of their legality seems to trouble only a few. In other words, the anti-trust laws are being gradually nullified by a development in American industry which no mere law can ever stop. Except for an occasional outbreak of denunciation from a Congressman seeking publicity we hear surprisingly little adverse criticism of the merger movement among the people.

Actually the desire to consolidate has not sprung unheralded from the brain of industry. Economic forces at work beneath the surface of commerce and trade have been preparing for the condition which we now suddenly find prevailing and which may seem to have developed overnight. But it is not so surprising to anyone who has studied the underlying motives that have led up to this eventful era, when all industry seems possessed with

*A bulletin issued by the Textile Division of the Department of Commerce.

the urge to combine into a few powerful units. To the outside observer, the so-called "man in the street," it has no doubt appeared as though a great magnet has been touched here and there to a scattered myriad of iron filings. The sweep of the merger magnet has left in its path some amazing combinations of industrial interests. A large food trust, mergers of national banking interests, department stores—almost any type of enterprise is open to infection from the merger germ. But neither the number of recent consolidations nor in some cases their unique nature has been unexpected among those who have, as we say, watched the forces at work beneath the smoke of manufacturing industry.

One of the primary forces responsible for an era of trusts and mergers is, in our opinion, the trade association. Apparently an innocent and quite praiseworthy affair, the trade association has probably laid the groundwork for many of the new combines. It has acted as the medium or catalyst for consolidations of several small and often unprofitable ventures into one heavily capitalized unit.

When trade associations first took form it is unlikely that many foresaw this outcome of the movement. Trade groups were merely regarded as an effective means by which an industry could regulate its output, protect its members, standardize its raw materials and products. Who ever thought that strong pools of capital would grow from these friendly contacts among members of a trade? From these informal gatherings of committees and these dignified by-laws and codes of ethics which have marked the visible activity of your ordinary trade group?

But the association movement, once under way, swept like a prairie fire in the wind through the ranks of industry. Never before had there been such a general willingness on the part of individuals and private organizations to unite in working for the constructive upbuilding of our industry and trade. A census of commercial and industrial organizations issued this year by the Department of Commerce listed the names of more than 13,000 associations. Back in 1912 the total number of listings was only 3,200. Even by 1926 there had been formed not more than 9,000 or about 55 per cent of the number shown in this year's census. Practically every branch of business activity is represented to-day by one or more associations. As the Commerce Department stated on the occasion of issuing its latest manual, the exchange of ideas and knowledge among competitors would have been considered commercial suicide twenty-five years ago, while it is now deemed beneficial and constructive—a distinct contribution to higher business ethics.

But the fact is now disclosed to a world of somewhat bewildered industrialists that ethics and information were not to be the final consequences of this association idea. The captains of industry were not content merely to sit together upon committees of standardization, nor to draft high-sounding by-laws and principles of trade. They were ambitious, it now appears, to sit upon directing boards of mergers and to draft plans for increasing the business of huge combines.

The trade associations will doubtless continue to multiply. What will come out of the merger movement, on the other hand, cannot be so easily predicted. There is fear in certain quarters that these new mergers are, after all, just as dangerous as the trusts of yore upon which Roosevelt and Sherman fell with battle axes of public indignation. But this fear is not widespread. The general feeling seems to be that the public is due for an era of good intentions just as soon as these new consolidations begin to fall over each other in an effort to serve their nation-wide family of consumers. The real conflict will be among the combines themselves. It may wax bloody before it subsides, for the stakes are tremendous and the financial power wielded by many of the mergers recently formed is almost without limit.

The small, independent factories see only storm clouds rolling over their heads, and in the end many of them will probably scurry to cover in the face of impending disaster by forming combinations of their own.

One of the evils of closely united capital and industrial resources lies in the domination of two or three strong companies within a trade association. Originally the business groups and organizations were formed to help their members by a co-operative exchange of experience and a fraternal spirit of "one for all and all for one." But with mergers among some of the larger members in a group there may easily be ushered in a kind of monarchy or dictatorship that will defeat the object of group co-operation. The only remedy for this evil would seem to be a general revolt staged by the smaller members, a walk-out and the subsequent formation of a new trade group.

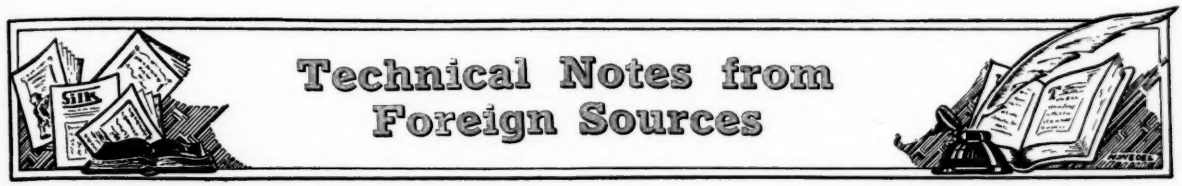
PLANS FOR HERCULES RESEARCH LABORATORIES UNDER WAY

Plans for the erection near Wilmington, Del., of a new station consisting of experimental and chemical research laboratories were announced a short while ago by the officials of the Hercules Powder Company.

Property consisting of about 300 acres is being acquired by the Hercules organization two and a half miles west of Wilmington city limits. Following the erection of the structures now being designed, the experimental staff and the research equipment will be moved there from Kenvil, N. J., the present site.

Construction will consist of a main building, containing chemical laboratories, offices and library, and auxiliary buildings housing experimental plants. A power-house and store rooms will also be erected. At the present station about one hundred and twenty people are employed, of whom more than half are technically trained chemists, physicists and engineers.

Removal of the Hercules experimental work to Wilmington will bring it into closer contact with the main office organization.



Mordanting and Dyeing of Cellulose Esters

British Patent No. 287,204; *Henry Dreyfus.*—Salts of suitable metals for mordanting, of aliphatic acids, are recommended; e. g., aluminium acetate, followed by a bath of weak sodium carbonate.

Dyestuffs for Viscose, Etc.

British Patent No. 287,609; *British Dyestuffs Corp., Ltd., and J. Baddiley.*—Secondary dis-azo dyestuffs are proposed, which consist of an aromatic nitro-amino-sulphonic or carbonic acid, diazotized and coupled to any suitable second component containing a free amino group, which in the mono-azo dyestuff so produced is further diazotized and the product coupled to an amino-naphthol-sulphonic acid or a derivative thereof, substituted in the amino-group. 2-Amino-naphthol-8-sulphonic acids and their derivatives are excepted. The products dye viscose in blue tones, well leveled.

Heating Processes Electrically

Electric Heat in Industry. Public Service Company of Northern Illinois, Chicago, Ill. 33 pages, illustrated.

A foreword to this interesting brochure declares that industries everywhere are adopting electric heat in order to profit by more efficient production methods. The booklet shows in pictures the partial extent of this trend towards electric heat in northern Illinois, and should be of some interest to textile processing men.

Dyeing of Mixed Weaves Containing Immunized Cotton

British Patent No. 268,327; *Etab. Petitdidier, Paris.*—The goods are first dyed with a dyestuff which will dye the immunized cotton, leaving the other material, e. g., viscose, untouched. After washing, the goods are treated with a dilute hydrosulphite bath, containing, e. g., 0.5 g. of hydrosulphite per liter of water, at 65° C., to render the non-immunized material perfectly white.

Mordanting of Cellulose Esters

British Patent No. 273,693.—The mordanting action of mordant, as, for example, chromium acetate, ferric acetate, aluminium acetate, is accompanied by a swelling action upon the fiber through presence in the bath of furfurol or other similar swelling agent. In the example given, the bath is distinctly acid with acetic acid; temperature of working, 60° to 70° C. After treatment, the goods are washed. In mixed weaves the swelling agents do not, of course, interfere with the simple mordanting of the non-cellulose-ester constituent.

Dyeing or Printing of Cellulose Esters or Mixtures Containing Them

British Patent No. 292,180; *Henry Dreyfus.*—Azo dyestuffs are proposed, whose first component is substituted by a side-chain containing one or more hydroxyl groups. E. g., p-nitro-aniline is condensed with chlor-butylene glycol, the nitro-group of the product reduced, the new amino-compound diazotized, and coupled to alpha-naphthylamine.

Fixation of Acid or Basic Dyestuffs

British Patent No. 283,281; *I. G. Farbenindustrie A.-G.*—The fabric or material, which may be cotton or viscose or cellulose esters, is treated with a complex tungstic or molybdic acid, this agent fixed upon the fiber, and the goods dyed. Cotton, for example, is treated with a 1% solution of benzidine hydrochloride, and after squeezing treated with a 1% solution of phospho-tungsto-molybdic acid; after rinsing, the goods are dyed.

Mordanting of Animal or Vegetable Fibers

British Patent No. 269,917; *Chem. Fabr. Milch A.-G.*—The agents proposed are the metallic salts (metals thereof are the usual "mordanting metals," aluminium, chromium, iron, tin) of cyclic sulphonic sulphonic acids heavily substituted by an aliphatic side-chain, such as palmito-benzene-sulphonic acid, stearo-toluene-sulphonic acid, isopropyl-naphthalene-sulphonic acid (already known as wetting-out agents). By use of such salts the fiber can be mordanted just as it comes from spinning or weaving, without previous removal of oily or greasy matter. Or, the alkali salts of such acids can be added to the ordinary mordanting solution. If alcohols, as methyl or ethyl alcohol, glycol, glycerol, etc., be also added to the bath, mordanting takes place even more effectively.

Developed Dyeings

British Patent No. 295,977.—The fiber, cellulose, either cotton or regenerated cellulose, is treated before weaving with an impregnating bath containing one of the Naphthol AS series. Warp and woof may be treated with a different Naphthol, of such nature that on developing the woven fabric as usual contrasting color-effects are produced as desired. Or, only one element of the weave may be so pre-treated, the other thus remaining undyed.

Sulphur Dyestuffs Fast to Chlorine

German Patent No. 432,177; *Kalle u. Co.*—By fusing hydroquinones or their derivatives with nitrocarbazoles, nitro-nitrosocarbazoles, aminocarbazoles, or any of their

derivatives, together with alkaline polysulphides, preferably under pressure, e. g., the products may be purified by extraction with amyl alcohol or nitrobenzene. The products dye cotton in tones ranging through blue to green and black.

Black Sulphur Dyestuffs

German Patent No. 445,890; *Comp. Nat. de Mat. Col et Manuf. Etc., Kuhlman, Paris.*—Hydroxylated naphthalenes, as beta-naphthol, 2-7-dihydroxy-naphthalene, and beta-dinaphthol, are first treated in the heat with anhydrous aluminium chloride, and the condensation products formed then heated with alkaline polysulphides as usual. The products must be free from dihydroxy-perylenes. The dyestuffs are in some cases fast to chlorine.

Discharge Printing

German Patent No. 435,734; *Chem. Fabr. Pyrgos u. Dr. Haller.*—In the common process for printing white discharges upon an Indigo ground, the pattern is printed upon the goods (dyed) with a paste containing an organic acid, the goods being then passed through a hypochlorite bath. By this method the fiber is liable to be greatly damaged where the bleaching takes place. The method proposed involves the use of the alkali salts of toluene-sulphon-chloramide as the bleaching agent, and the use of a catalyst, such as a salt of copper, iron, cobalt, nickel, vanadium, etc. The goods are printed with a paste containing an organic acid, such as lactic acid, and the catalyst, such as copper sulphide, added in the form of paste. After drying, the goods are passed through a hot 10% bath of the sodium salt of toluene-sulphone-chloramide. In another example, the acid used is tartaric, and the catalyst is ferric chloride.

Vat Dyestuffs of the Anthraquinone Series

German Patent No. 432,579; *I. G. Farbenindustrie A.-G.*—Compounds in some sense analogous to the Algol Yellows, or benzoylated alpha-amino-anthraquinones. The anthraquinone derivatives used must contain at least one amino-group in the alpha position. The introduced group is a beta-naphthol radical containing no free hydroxyl group. If an alpha-naphthol residue is introduced, the properties of the dyestuff are in general not different from the benzolated product. The beta-naphthol derivatives, on the other hand, display greater affinity for vegetable fibers than do the benzoylated products. Some of the new dyestuffs can be best dyed from a hot vat, in distinction from the simpler benzoylated or dibenzoylated homologues. This fact makes it possible to employ these new dyestuffs in mixture with some of the Indanthrene Blues, for example, which are dyed from a hot vat.

Combination of Anthraquinonoid and Indigoid Dyestuffs in Printing

Prof. Dr. Haller; Mell. Textil-Ber. 9, 924 (1928).—As far back as in 1912 the author found that a common Indigo-Rongalite-discharge-color could be combined with ferrous sulphate and tin salt, and that it was therefore directly possible to proceed to the synthesis of a color discharge which would enable one to use the well-known fast anthraquinonoid dyestuffs as discharge colors on the indigoid dyestuff ground. This procedure was not, *a priori,* to have been supposed possible. The author, before the carrying out practically of the above idea, had undertaken a large number of experiments on the preparation of iron and of tin hydrosulphites, so as to use such substances as discharges for the indigo ground, as well as fixing agents for anthraquinonoid dyestuffs present, by a passage through hot concentrated caustic—analogous to the Jeanmair procedure. The conversion of this idea to a practical process went to pieces, so to say, on the excessive instability of the substances involved.

Therefore, it was all the more surprising to observe that a combination of formaldehyde-sulphoxylates with Leucotrope W, and then ferrous sulphate and tin salt, proved feasible within the thickening paste without any special conditions being necessary, and that such a printing paste showed actually a high degree of stability, so that it was possible with confidence, on the basis of this observation, to construct, so to speak, a color discharge procedure for indigoid grounds, which would make possible the turning out of a fast product, which up to that time had not been attained. As a matter of fact, a printing paste of the following composition: 112 g. Starch gum thickener, 12 g. Rongalite C, 32 g. Rongalite CL, 8 g. Anthraquinone 30% paste, 20 g. water, 16 g. zinc oxide, 15 g. Indanthrene Yellow R, Double Paste, 30 g. ferrous sulphate, 5 g. tin salt, 70 g. Gum Senegal, printed upon indigo-dyed goods, steamed, and passed for 18 to 20 seconds through a 20° Be. caustic soda bath at 80° C., gave excellent yellow discharges upon a blue ground. Thus, it naturally became possible to devise a whole series of combinations, by using blue and violet anthraquinonoid dyestuffs; and also a lively green was within reach, by employing a mixture of blue and yellow indanthrenes. As a fact, this discharge procedure has proved excellent in practice, and has made it possible to imitate the old printed blue goods, yellow-green, two-blue-white, or two-blue, in handsomer, livelier, and, above all, faster quality.

The original, timid idea, that the formation of a vat, which might possibly take place, could dull the discharge effects in the alkaline aftertreatment, showed itself to be without foundation, particularly if one took the precaution of adding a little manganese dioxide paste to the caustic bath. Likewise, the high concentration of the hot caustic liquor showed itself to occasion no cause for apprehension; in fact, a welcome improvement of the fastness to rubbing of the indigo ground was evident after this alkaline treatment.

It straightway seemed of reasonable promise to employ the new procedure not only upon an indigoid ground, but to extend it to all possible indigoid dyeings which could be discharged with sulphoxylates, so that one could

obtain a variety in the design color as well as in the ground color. And again, in fact, very handsome effects were successfully obtained upon ground colors produced with the different Ciba dyestuffs, Ciba Blue 2B, Ciba Violet B, Ciba Orange G, and Ciba Pink B, with the use of certain anthraquinonoid dyestuffs. Very fast results were obtained, particularly more fast to rubbing than in the case of the indigo ground. The method is as follows:

The goods, mercerized or unmercerized, are first dyed as usual in the jig with the different Ciba colors. For a ground of Ciba Blue 2B, for example, a bath of 200 liters for one piece of 120 meters, equal 10 kg., is previously prepared with 100 c.c. of caustic soda of 36° Be., and 50 g. hydrosulphite conc. powder, the goods wetted out in this for three passages, then the following vat added, divided between three passages; 200 g. Ciba Blue 2B powder, 20 g. Turkey Red oil or Solapol Oil, 20 liters boiling water, 600 c.c. caustic soda of 36° Be., 500 g. hydrosulphite conc. powder.

The goods are dyed then for two passages at 50° C., then for eight at 60° C., the bath then run off, and the goods aired for four to six passages, rinsed in slightly acidified water, and then in fresh water. Ciba Violet B powder is used in exactly the same way. The dyed goods are now printed with the proper discharge color. In contrast to the original procedure already given, it has proved best to prepare a stock discharge paste, and to add to it the desired design color. Thus, the stock discharge paste is prepared as follows: 7.1 kg. white discharge, 0.5 kg. ferrous sulphate, 0.2 kg. tin salt, 0.2 kg. glycerol. The white discharge paste consists of 12 kg. British gum in powder, 3.5 kg. glucose, 10 kg. water, 10 kg. Rongalite CL, 3 to 6 kg. zinc oxide, 3 kg. water, 5 kg. Leucotrope W, 9.5 kg. rape-seed oil, 0.5 kg. oil of turpentine, 1.8 kg. gum 1-1.

The color discharge consists of 600 g. Cibanone Blue GCDN double in paste, 600 g. Cibanone Blue RSN double in paste, 8,700 g. stock discharge, 500 g. Rongalite CL.

This discharge color is printed upon the previously dyed goods in combination, as in the swatches (four of them are given as inserts in the original), or with Aniline Black, or with a normal hydrosulphite white, and steaming is carried out as usual in the Mather-Platt. Then follows the passage of the goods through the concentrated caustic soda bath. This bath is used in an iron roller vat of about 500 liters capacity. The goods should remain immersed in the lye for 15 to 18 seconds. The bath is heated to 80° to 90° with indirect steam. From the caustic soda bath the goods, after thorough squeezing, pass into a vat of hot water, in which the actual removal of the condensation product of the leuco dyestuff with the Leucotrope W takes place. After the passage through this bath of dilute caustic (made so, of course, by the caustic liquor carried over from the preceding bath) the goods pass into another bath of hot water, then into one of hot sulphuric acid of about 8° to 10° Be., and are finally well washed, and soaped full width before drying.

As a green discharge for the different grounds the following may be recommended: 55 g. Cibanone Blue 3G, 350 g. Anthraflavine in paste, 700 g. stock discharge, 25 g. Rongalite CL.

Making up is carried out in the above-directed way. Naturally the design colors can be combined with black or white effects, or with the two together. As black, Aniline Black can be employed without difficulty; for white, the well-known Leucotrope W sulphoxylate discharges are used.

So, the possibility exists, of producing an extraordinarily solid dyeing, fast to air, light, water and washing, which in degree of fastness can be surpassed only if one uses Cibanone dyestuffs for both ground and pattern—which at present hardly seems possible. By the reserve method, polychrome prints on Cibanone grounds with the ordinary printing machines can hardly be produced, although methods are known of printing Cibanone reserves under Cibanone grounds. But, at all events, the combination of indigoid and anthraquinonoid dyestuffs for colored prints means a long step forward toward the ideal of fastness, little to be looked for even in straight dyeing, much less in printing.

Blueing in Cotton Bleaching

Richard Huenlich; Mell. Textil-Ber. 9, 928 (1928).—The author, in his brief discussion, emphasizes the fact that "white" covers as many shades as "black," in the sense in which the latter word is used as a designation for a color. Beside goods which are actually a clean, pure, unshaded white, there are shades of yellowish and of bluish white which occur by choice or of necessity. "The concept of white," he says, "extends beyond mere pure white, as the stock in large warehouses can prove. In the case of goods which come from different bleacheries, differences can, of course, be observed on comparison of different lots. A single lot, examined by itself, appears pure white. If, in the process of bleaching, the chlorine bath is allowed to act too long upon the goods, a decided yellow tone is obtained, and the uneven results visible in a series of lots, according to the manner and degree of bleaching, necessitates the use of differently shaded degrees of blueing. The use of blueing agents must involve good judgment, for too much of a blue shade gives a poor impression on inspection of the goods, and suggests that there is involved an attempt to improve poorly bleached material. The purer and more natural the white is, the finer the appearance; a white shading toward greenish (blueing of yellowish goods) spoils the appearance of the goods. The more yellow the bleached goods the redder must be the tone of the blue used, if a true, pure white is to be obtained."

Glucose Procedure for Indigo Vat Dyeing

Prof. M. Tschilikin; Mell. Textil-Ber. 9, 926 (1928).—An interesting, highly technical discussion of the method.

Increasing of Affinity of Animal Fibers for Dyestuffs

German Patent No. 448,797; *Chem. Fabr. vorm. Sandoz, Basle.*—By esterifying the material with the halogen derivatives of aliphatic, aromatic, or hydro-aromatic carboxylic or sulphonic acids, or the anhydrides of these, the material is made more capable of taking up either acid or basic dyestuffs; in the case of direct colors also, wool, e. g., can be dyed in a neutral bath without any special additions. The bath is also exhausted more rapidly than is the case with unesterified wool.

According to one (out of several) example, 20 g. of wool are worked thoroughly at 40° to 50° C. for 2 hours in 250 g. of a 20% solution of p-toluene-sulphon-chloride in toluene, then squeezed, and well washed, at 60° to 70°, in three successive soap baths, containing 0.3, 0.2, and 0.1 g., respectively, of soap. The material is then washed for some time in cold water and dried.

GENERAL DYESTUFF ISSUES CARDS SHOWING NEW COLORS

The General Dyestuff Corporation has recently given to the trade several circulars illustrative of new dyes for which they are the agent. Among these are: Indanthren Olive 3G Paste and Powder, Indanthren Brown 3GT Paste and Powder, Katigen Bordeaux B. A new product, Raynise K, manufactured by the I. G., and Fast Red Salt RL were also described.

It is claimed that the new Katigen Bordeaux (manufactured by the General Aniline Works, Inc.) is a sulphur color of particular interest on account of its bright claret shade, possessing excellent solubility and fastness to light, washing, cross dyeing, level dyeing, and also exhausts well.

Raynise K is recommended for sizing of rayon cloth. The General Dyestuff claims that this material retains its luster and is not stained, that the threads are remarkably smooth and do not stick together, and the tensile strength is increased about 35%. They maintain that it may also be used to improve the winding quality of dyed rayon, and that it is useful in the finishing of rayon or rayon mixed piece goods, giving an excellent feel without impairing the luster.

The new stable Diazo salt, Fast Red Salt RL, so states the booklet describing it, produces yellowish and bluish reds on Naphthol AS-RL ground and on Naphthol AS-D ground, respectively, and has a very good fastness to light, washing and chlorine.

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i. e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

FINISHER WANTED

Finishing plan located in New Jersey has an opening for an experienced finisher of cotton and rayon fabrics. Only men with experience and proven ability in producing finishes need apply. Commensurate salary. Address: Classified Box 558, American Dyestuff Reporter.

ASSISTANT TO MANAGER WANTED

An assistant to the manager of an important dyeing, weighting, printing and finishing plant, located in the vicinity of New York City, handling silk, rayon, Celanese and mixtures. This opportunity is open for a graduate chemist, 35 to 45 years old, thoroughly familiar with all processes of dyeing, weighting, printing and finishing, having at least seven years' practical experience in these lines in a plant handling a minimum of one thousand pieces per day. The applicant should also be able to supervise research laboratory. Knowledge of cloth construction desirable. Apply by letter, stating qualifications and fully outlining past experience. Also give references. All replies will be held as strictly confidential. As this is a very unusual opportunity to become one of the executives of a large and growing organization, only a very high type technician need apply. Address: Classified Box 553, American Dyestuff Reporter.

CHEMIST-COLORIST WANTED

Chemist-Colorist, experienced in textile printing, desires position. Address: Classified Box 554, American Dyestuff Reporter.

POSITION WANTED

Expert dyer on rayon knit goods; many years' practical experience dyeing tubular rayon cloth (tricolettes); five years with last concern as boss dyer; capable of taking complete charge of dyehouse. Excellent references. Address: Classified Box 555, American Dyestuff Reporter.

TEXTILE MILL OPENING

Large textile mill has opening for a man thoroughly familiar with stripping of rags and shoddy. Good opportunity for advancement. Address: Classified Box 556, American Dyestuff Reporter.

JUNIOR TEXTILE CHEMIST WANTED

Wanted—Junior textile chemist by ladies' dress manufacturer doing screen printing. Should have knowledge of application of all kinds of cotton colors, wools and silks. Capable of doing routine and development work. Textile school graduate preferred. State experience and salary expected. Address: Classified Box 557, American Dyestuff Reporter.